

164878 K-3



PROJECT
1526892

VOLUME IIIb OF III

PLANNING DOCUMENTS

QUALITY ASSURANCE PROJECT PLAN

FINAL

REMEDIAL INVESTIGATION/
FEASIBILITY STUDY

BELOIT CORPORATION
ROCKTON FACILITY
ROCKTON, ILLNOIS

MAY 1994

PREPARED FOR:
BELOIT CORPORATION
ROCKTON, ILLINOIS

...
PREPARED BY:
WARZYN INC.
MADISON, WISCONSIN



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FIELD SAMPLING PLAN (FSP)

The field activities for Phase 2 at the Beloit Corporation Rockton facility RI/FS will follow the approved June 1992 Field Sampling Plan from the QAPP (included here), as modified by the Phase II Work Plan Addendum, to be approved by the Illinois Environmental Protection Agency.

PROJECT
1526800

FIELD SAMPLING PLAN
REMEDIAL INVESTIGATION/FEASIBILITY STUDY

**BELOIT CORPORATION
ROCKTON FACILITY
ROCKTON, ILLINOIS**

JUNE 1992

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REMEDIAL INVESTIGATION/FEASIBILITY STUDY

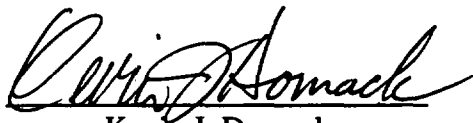
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WARZYN INC.
MADISON, WISCONSIN


Kevin J. Domack
Project Manager

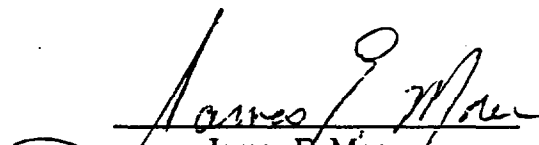

James E. Moser
RI Task Manager

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- A-A6 - Calibration and Maintenance of the Flame-Ionization Detector

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OBJECTIVES

The primary objective of the activities described in the Field Sampling Plan (FSP) is to obtain representative data for use during the Remedial Investigation/Feasibility Study (RI/FS) analysis. Sampling activities described in the FSP will be performed during the site investigation activities of the RI. Documentation procedures for sampling activities are described in the Data Management Plan (Work Plan, Appendix A).

The RI will focus on the definition of potential sources of soil or groundwater contamination, determine the nature and extent of contamination and collect data for the Baseline Risk Assessment and Feasibility Study.

The RI is designed to be implemented as an integrated investigation. Data collected during initial RI activities will be used to determine appropriate sampling activities and monitoring locations for subsequent activities of the RI. During initial RI activities, potential contaminant source areas will be identified and chemically characterized (Source Characterization), and potential contaminant migration pathways will be evaluated (Migration Pathway Assessment). Data generated from the Source Characterization and the Migration Pathway Assessment may be used in conjunction with data from the Site Evaluation Report (SER) to evaluate the extent and magnitude of contamination along the potential migration pathways. The magnitude and extent of contamination will be determined during Contaminant Characterization, by sampling media of concern.

Source Characterization data use objectives include:

- Identify the location and extent of potential contaminant source areas; and
- Characterize the chemical and physical aspects of potential contaminant source areas and contaminant source materials.

Migration Pathway Assessment data use objectives include:

- Identify the most likely pathways for contaminant migration;
- Characterize the hydrogeologic and hydrologic setting of the site; and

- Describe the characteristics of soils and sediments in the site area.

Contaminant Characterization data use objectives include:

- Describe the magnitude of groundwater, surface soil, and subsurface soil contamination; and
- Evaluate the extent of contaminant migration along potential migration pathways at the site.

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SCOPE OF SAMPLING ACTIVITIES

The FSP describes the procedures and practices for use in performing site investigation activities during the RI/FS. These procedures include a description of the sample designation system, personnel and their responsibilities, and the sampling methods to be used during RI activities. The scope of RI activities discussed in the FSP include:

- soil gas sampling;
- shallow soil borings and soil sampling;
- geophysical surveying (surface and downhole);
- site mapping;
- evaluation of existing wells;
- deep and intermediate groundwater quality borings and groundwater sampling;
- monitoring well installation/replacement and soil sampling;
- existing monitoring well and standpipe abandonment;
- hydraulic conductivity testing;
- water level measurements;
- surface water and sediment investigation;
- surface soil assessment;
- air assessment;
- ecological assessment; and
- groundwater sampling and chemical analysis.

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SAMPLING LOCATIONS AND NUMBERS OF SAMPLES

3.1 SOURCE CHARACTERIZATION

Source characterization will include soil gas sampling, shallow soil borings and soil sampling, and a electromagnetic (EM)/magnetometer surface geophysical survey.

3.1.1 Soil Gas Survey

A soil gas survey will be performed in known suspected contaminant source areas as well as areas where potential sources may exist. The objective of this survey is to locate potential sources of volatile organic compounds (VOCs). Previous investigations have indicated that several potential source areas may exist. However, other identified source areas may exist in the site vicinity. Drawing 15268-6 shows the proposed locations for the soil gas survey. Approximately 100 samples, ten field duplicate samples, and one field blank per day will be collected. Each sample will be analyzed by an in-field, portable gas chromatograph (GC) for target VOCs (Method EPA 8010/8020).

3.1.2 Shallow Soil Borings and Soil Sampling

Twelve shallow soil borings (SB8 through SB19) will be performed to identify the presence of VOCs in potential source areas. The borings will be drilled to a minimum depth of the water table (anticipated depth 25 to 35 ft). The proposed soil boring locations (refer to Drawing 15268-7) have been selected based on information gathered during the preparation of the SER. Modifications or additional soil boring locations may be established, pending the results of the soil gas and surface geophysical survey (see Section 5 of the Work Plan for coordination between Warzyn and IEPA). Soil samples will be collected at 2.0 ft intervals using a 3-in. split spoon sampler. The 3 in. sampler is primarily needed for acquiring enough sample volume to accommodate splitting samples with the IEPA. However, in some instances, a 2 in. split spoon sampler may be necessary for soil composition evaluation. Field decisions will be required to determine which sampler is most appropriate for each boring. A soil boring log describing the materials encountered, visual evidence of contamination, and PID screening results will be recorded by the field geologist/engineer. Soil samples representative of each unique lithology will be retained in 8-oz soil sample jars.

A sufficient quantity of representative soils will be collected from each split spoon sample to fill, at a minimum, one 8-oz. wide mouth glass jar for potential semi-volatile analysis, one 8-oz wide mouth glass jar for potential pesticide/PCB analysis, one 8-oz wide mouth glass jar for potential metals and cyanide analysis, and two 4-oz. wide mouth glass jars for potential VOCs analysis, and to partially fill one 8-oz. wide mouth glass jar for field headspace analysis. Vertical split samples will be collected from the split spoon samples by cutting the soil sample in half (parallel with the long axis of the split spoon sampler) and collecting samples, top to bottom, from each half of the split spoon device. One half of the split spoon sample will be used to fill the 4-oz. sample jars immediately after opening the split spoon. The other half will be used to log the borehole and fill the four 8-oz. soil sample jars one of which is to be used for field headspace analysis. The field headspace method is described in Section 5.2.3 of the FSP.

Surface soil samples (6 in. depth) will be collected from each shallow boring location and analyzed, for a total of 12 samples. Additional surface soil samples will be collected from an off-site location for use as a background surface soil sample. Surface soils will be analyzed for U.S. EPA CLP Target Compound List (TCL) organics, and Target Analyte List (TAL) inorganics.

Twenty-four sets of subsurface soil samples will be selected for U.S. EPA CLP analysis based on the following criteria:

- Detectable PID (11.7 eV) or FID readings greater than background;
- Visual evidence of soil contamination; and
- Distinct soil stratigraphic changes (e.g., contact at aquifer/aquitard boundary).

Selection of subsurface soil samples for analysis will be determined in the field and based on in-field screening results. During sample collection, subsurface soils will be visually examined and screened for VOCs with a PID/FID. Subsurface soil samples with obvious visual contamination and/or detectable PID/FID field headspace readings will be selected for U.S. EPA CLP analysis. If no indications of VOC contamination are observed using in-field techniques, at least one set of subsurface soil samples will be collected from each boring for U.S. EPA CLP analysis using the following three criteria:

- At a distinct soil stratigraphic change;
- At a depth of 10 ft for borings in areas with low soil gas readings; or
- At a depth of 5 ft for borings in areas with high soil gas readings.

A minimum of 24 sets of subsurface soil samples (an average of two samples per soil boring) will be analyzed for U.S. EPA CLP parameters. At least one set of soil samples will be collected from each boring for analysis.

3.1.3 Electromagnetic/Magnetometer Survey

An electromagnetic ground conductivity meter and/or a proton precession Magnetometer will be used to perform a magnetic survey to locate potential buried metallic objects such as drums and underground storage tanks. The surveys will be performed on and around the 1314 Watts Avenue and Soterion properties in the Blackhawk Acres Subdivision (refer to Drawing 15268-6). Each instrument will be calibrated prior to each use as described in the users manual. The initial survey will collect readings on a profile at 10-ft centers in the areas of interest. A coarser grid (20 ft centers) may be employed in areas surrounding these properties. During the initial survey, anomalous areas will be located on a field map and marked with pin flags in the field. Subsequently, a second survey will be performed over the anomalous areas using a tighter grid (5-ft centers). Quality assurance will be effected by the duplication of randomly selected survey measurements to examine instrument response.

3.2 MIGRATION PATHWAY ASSESSMENT

3.2.1 Hydrogeologic Investigation

3.2.1.1 Site Mapping and Evaluation of Existing Monitoring Wells - A topographic map with a scale of 1 in. to 200 ft and a contour interval of 2 ft has been prepared for use in the RI/FS (Drawing 15268-7). This base map will be used to develop other drawings and maps showing RI data.

An inventory and evaluation of existing monitoring wells will be performed to assess their condition. This will include both IEPA and Beloit Corporation wells. The following will be evaluated:

- well location
- integrity of protective casing (to include cap, lock, and identification) and the surficial seal
- integrity of well casing and screen
- water level
- total well depth
- amount of sediment in well

Wells will be developed, if necessary, to provide representative water level measurements using procedures described in the next to last paragraph of subsection 5.4.3.2 of the FSP. Functional PVC and stainless steel wells will be included in the water level measurement task and functional stainless steel wells will be included in groundwater quality sampling activities. Wells which are determined to be non-functional will be properly abandoned according to IEPA monitoring well abandonment procedures (IEPA 1990) presented in paragraph two of Section 5.4.3.2 of the FSP.

Natural gamma ray logging will be conducted in existing functional wells for use in evaluating subsurface stratigraphic conditions. At locations of a well nest, only the deep well will be natural gamma ray logged.

3.2.1.2 Deep and Intermediate Groundwater Quality Borings and Groundwater Sampling - Five deep soil borings (DB1, DB2, DB3, DB4, and W22C) and two intermediate soil borings (W23B and IB1) will be performed within the RI/FS boundaries. The deep borings will be drilled to a minimum depth of 100 ft and the intermediate borings to a minimum depth of 70 ft at the proposed locations shown on Drawing 15268-7. Total actual depth is to be determined in the field based on stratigraphy and groundwater sample screening results using a field GC. Drilling and groundwater sampling will be discontinued at a boring when one of the following situations develops:

- When groundwater screening results indicate that VOCs are not present and the 100 ft (for deep borings) or 70 ft (for intermediate borings) minimum depth has been reached, sampled, and analyzed;
- When groundwater screening results indicate that VOCs are present and the minimum depth requirement of 100 ft (deep borings) or 70 ft (intermediate borings) has been achieved, and a minimum of two consecutive clean groundwater samples have been collected below the deepest VOC detection in that boring; or
- For intermediate borings at potential source areas, and deep borings where non-aqueous phase liquids have been identified, when a confining unit greater than or equal to 3 ft in thickness is encountered, irrespective of depth. In this situation, the last groundwater screening sample will be collected at the top of the confining unit.

None of these borings will be advanced into bedrock during the Phase 1 investigation.

Dual-tube reverse circulation drilling methods (Appendix A - A2), will be used to advance the deep groundwater quality borings. Plugged, screened hollow-stem augers will be used to advance the intermediate groundwater quality borings. During drilling, groundwater samples will be collected every 10 ft, beginning at the water table. Groundwater samples will be collected through the center of the drill string or casing with a bailer, or submersible pump. Groundwater samples collected from the seven borings will be analyzed for total VOCs with an in-field gas chromatograph (GC). The GC data from the groundwater samples will be used to obtain a vertical representation of the groundwater quality beneath the site.

Upon completion of the borehole, downhole geophysical logging will be performed to obtain a natural gamma ray log. The natural gamma ray log of the borehole will be used in conjunction with cuttings for subsurface interpretation of soil stratigraphy and assessment of potential subsurface preferred contaminant migration pathways.

Monitoring wells will be installed in those borings where the GC results indicate the presence of VOCs. Screen placement will be determined based on GC results, soil stratigraphy, and gamma ray response.

One deep geotechnical boring (GB1) will be advanced to approximately 100 ft using mud rotary drilling techniques. This boring will be used to ground truth the gamma ray logging tool and to provide a representative_{on} stratigraphic sequence for the site. Methods are described in Section 5.4.3.

After completion of downhole logging of the borehole, any borehole not completed with a well, will be abandoned in accordance with IEPAs guidance document - Groundwater Monitoring Network for Non-Hazardous Solid Waste Disposal Facilities (1990). These abandonment procedures are detailed in Section 5.4.5.2 of the FSP.

3.2.1.3 Monitoring Well Installation/Replacement and Soil Sampling -

Initially, twelve new wells will be installed (nine on Beloit Corporation property), including six water table wells and six piezometers. One well (W24) will be installed hydraulically upgradient of the site adjacent to IEPA monitoring well G107, to assess background groundwater quality. Existing monitoring wells W3, W5, W8, and W11 will be replaced. Additional monitoring wells may be installed during the Phase 2 investigation. The location, number and type of wells to be installed will be determined from information obtained during the initial investigation (Phase 1) activities.

Monitoring wells will be installed using hollow stem auger, air rotary, or rotary wash drilling methods. Split-spoon soil samples will be collected every 2.5 ft during drilling of the boreholes. Soil samples representative of local stratigraphy will be saved in glass sample jars. Twenty of the collected soil samples will be submitted for grain size analysis by ASTM D422. Grain size distribution results will be used to confirm and enhance the visual description of soils made by the site geologist during drilling.

In addition to obtaining a stratigraphic log from split-spoon sampling, each of the monitoring well boreholes will be natural gamma ray logged. The natural gamma ray logs of the boreholes will provide a continuous record of variations in the natural radioactivity of the borehole and an indication of soil stratigraphy. This information will be used during evaluation of site geology, by indicating variations in clay content of the unconsolidated deposits at the site.

New monitoring wells will be developed by alternatively surging and purging the wells for a minimum of 30 min. using a bailer. After the surge and purge cycles are completed, the well will be pumped until ten well volumes are removed or until the pH and specific conductance of water removed from the well has stabilized.

Drill cuttings, drilling fluids (mud and water) and extraneous soils derived from split spoon sampling will be evaluated in the open atmosphere during drilling with an 11.7 eV PID in the field. If PID readings are equal to or exceed 5 ppm then these cuttings, soils and fluids, will be placed in 55 gal drums, labeled and retained on-site on Beloit Corporation property. If PID readings are less than 5 ppm then these cuttings, mud and extraneous soils will be disposed of on-site on Beloit Corporation property. TCLP analysis will be performed on composites of the containerized cuttings, mud and soils (PID \geq 5 ppm) in order to determine an appropriate disposal option. Disposal of water used as a drilling fluid is discussed below.

Development, drilling and purge waters will be separated based on the presence or absence of a non-aqueous phase liquid. Those development, drilling and purge waters containing a non-aqueous phase liquid will be considered a hazardous waste, drummed, stored separately and disposed of at an appropriate hazardous waste facility. Development, drilling and purge waters not containing a non-aqueous phase liquid will be disposed in the sanitary sewer along with decontamination water for treatment at the local POTW, assuming approval can be obtained from the POTW. If approval can not be obtained, these materials will also be drummed, stored in a secure area on Beloit Corporation property and disposed of at the time of the final remedy.

3.2.1.4 Existing Monitoring Well and Standpipe Abandonment - Four existing monitoring wells (W3, W5, W8, and W11) and seven standpipes (SP1, SP3, and SP8 through SP12) are to be abandoned during Phase 1 of the RI. They will be abandoned in accordance with IEPA's guidance document - Groundwater Monitoring Network for Non-Hazardous Solid Waste Disposal Facility (1990). These abandonment procedures are detailed in Section 5.4.3.2 of the FSP.

3.2.1.5 Hydraulic Conductivity Tests - Hydraulic conductivity tests will be performed in the new wells installed during the RI. Single well baildown test methods will be used and data analyzed using the Bouwer and Rice (1976) method. Additional baildown hydraulic conductivity testing may be performed in existing functional monitoring wells.

3.2.1.6 Water Level Measurements - A minimum of four rounds of water level measurements will be conducted during the RI. Water level measurements will be obtained with an electric water level indicator. One round of water levels will be measured following completion of the monitoring well installation. Water levels will be measured concurrent with groundwater sampling events. Bimonthly measurements will be conducted thereafter for a period of eight months, if necessary from the initial measurement event. The water level measuring device will be deconed between wells.

3.2.2 Surface Water and Sediment Investigation

Surface water drainage patterns will be identified and located based on field observations. These drainage patterns and runoff characteristics will be evaluated for the potential of erosional transport of contaminated surface soils off-site. It is anticipated that three staff gauges will be installed at the site (along the Rock River). Specific staff gauge locations will be determined based on field observations of optimum locations. Existing staff gauges at the site will be evaluated for integrity and monitoring use. Surface water level measurements will be collected concurrently with groundwater level measurements.

3.2.3 Surface Soil Assessment

The potential for exposure to contaminants via the surface soils pathway will be evaluated based on samples collected for chemical analysis from the 12 shallow borings (Drawing 15268-7).

3.2.4 Air Assessment

Wind patterns will be evaluated, based on available meteorologic information, to determine possible air pathway exposure scenarios. Air quality data collected from health and safety monitoring during site investigation activities will be used to assess the potential for release of contaminants into the air. Samples will not be collected for laboratory analysis.

3.2.5 Ecological Assessment

A preliminary ecological assessment of the site and adjacent areas will be performed. Ecological resources will be investigated by a site walkover by a field ecologist to note types and approximate sizes of plant communities of the site and adjacent undeveloped areas. Potential of the site and surrounding areas as habitat for vertebrate species will be assessed. Wetland areas will be classified according to the U.S. Fish and Wildlife Service (U.S. F&WS) system (Cowardin, et. al., 1979) and their sizes estimated by use of the Federal Manual for Identifying and Delineating Jurisdictional Wetlands (Federal Interagency Committee for Wetland Delineation, 1989).

Site observation will be supplemented by information available from site and federal agencies concerning the following ecological resources:

- Hydric soils (U.S. Soil Conservation Service)
- Rock River fishery (Illinois Department of Conservation)
- Threatened or endangered species (Illinois Department of Conservation and U.S. F&WS)
- Designated area wetlands (U.S. F&WS National Wetland Inventory Map)

A qualitative preliminary ecological assessment will be developed to evaluate potential contamination by site chemicals detected by RI sampling activities. Potential effects of these contaminants will be discussed for species likely to inhabit the site or areas affected by site contaminants. If a preliminary evaluation suggests a serious impact by the site on area natural resources, further investigation may be required (U.S. EPA Region V, Tasks 3-6, undated, unpublished memorandum).

3.3 CONTAMINANT CHARACTERIZATION

3.3.1 Groundwater Sampling

Groundwater samples will be collected from the following locations during the Round 1 sampling event:

- The 12 new groundwater monitoring wells installed during well installation activities of the RI (Refer to Drawing 15268-7);
- The four replacement wells W3, W5, W8, and W11;
- Existing monitoring wells W12 through W18 (7 wells), and G103S and D, G104, G107, G108S and D, G109, and G110 (8 wells); and
- Private wells as specified in the private well sampling document.

Groundwater sampling will be used to determine the chemical characteristics of groundwater contamination. Samples will be analyzed for the following chemical and physical parameters:

- U.S. EPA CLP TCL organics;
- U.S. EPA CLP TAL inorganics;
- Sulfate;
- Chloride;
- Alkalinity;
- Total phenolics;
- Nitrate-Nitrite nitrogen;
- Ammonia nitrogen;
- Total dissolved solids;
- Field pH; and
- Field specific conductance.

Inorganic analyses will be performed on filtered samples collected during Round 1, consistent with U.S. EPA Region V QA Directives. Round 2 samples will be analyzed for contaminants of concern based on the results of Round 1 sampling. Technical Memorandum No. 1 will describe analytical results and address data gaps requiring additional sampling and analysis to be performed during the Phase 2 investigation.

Appropriate sample containers, preservation, and packaging are described in Table 1-2 of the QAPP.

3.4 QUALITY ASSURANCE SAMPLING

Quality control samples will be collected during sampling activities associated with the chemical characterization of groundwater, surface soils, and subsurface soils. Quality control samples will consist of sample duplicates, field blanks, and matrix spike/matrix spike duplicate (MS/MSD) samples, as described below.

3.4.1 Field Blanks (FB)

For soil samples, no field blanks will be collected due to the unavailability of suitable blank material. For water samples, one field blank will be prepared for each container type and size. Field blanks will be prepared according to the following schedule for each sampling activity:

- One field blank for every 10 or fewer samples of water collected; and
- For each sample period, a minimum of one blank for each group of parameters per sample matrix.

The field blank samples will be prepared using deionized water stored in polyethylene containers. For monitoring well samples, the water will be routed through the sampling device before transfer to the container. Soil gas field blanks will be prepared by collecting a sample of ambient air after decontamination of the sampling system.

3.4.2 Trip Blanks (TB)

A trip blank for volatile organic analysis (VOA) will be included in each sample shipment containing water matrix samples for VOA. The trip blank will consist of two 40-ml VOA vials filled with deionized water with a Milli-Q cleanup. It will be prepared in the office or laboratory, transported to the field and shipped with the other samples to the designated laboratory without being opened. It will be packaged using standard procedures as for other sample bottles.

3.4.3 Matrix Spike and Matrix Spike Duplicates (MS/MSD)

For water samples, one sample per group of 20 or fewer samples collected for VOA and extractable organics analysis during each sampling activity will be selected for matrix spike/matrix spike duplicate (MS/MSD) analysis. For Base/Neutral Acids (BNAs) and Pesticides/PCBs, double the normal sample volume will be collected (i.e., four, 1-L bottles). For VOCs, triple the normal sample volume will be collected (i.e., six, 40 ml VOA vials).

3.4.4 Sample Duplicates

One duplicate sample will be collected for each increment of 10 or fewer samples collected for each matrix during each sampling period. A duplicate sample will consist of a sample obtained from the same sampling device as the original sample.

Due to the sample volume requirements and the soil volume obtained using split-spoon sampling methods, sample duplicates for subsurface soil samples may not be possible. Sample duplicates for subsurface soils will only be performed if the split-spoon sample volume is adequate during subsurface soil sampling procedures.

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SAMPLE DESIGNATION

A sample numbering system will be used to identify each investigative and quality control sample. Each sample identifier will include the project identifier code, sample type and location code, and a sampling event code. The sampler will maintain a log book containing the sample identification listings.

4.1 PROJECT IDENTIFIER CODE

A two-letter designation will be implemented to identify the sampling site. The project identifier will be "BC" to signify the Beloit Corporation Rockton Facility site investigation.

4.2 SAMPLE TYPE AND LOCATION CODE

Each sample collected will be identified by a two-letter code corresponding to the sample type. Sample type codes to be used for the various samples collected are:

- GD - groundwater sample from deep groundwater quality boring
- GI - groundwater sample from intermediate groundwater quality boring
- PW - groundwater from a private residential well or municipal well
- GW - groundwater sample from monitoring well
- SS - split spoon or subsurface soil sample
- SU - surface soil sample
- FB - field blank
- TB - trip blank

Other letter designators may be added for sample activities of later subtasks.

The location code will follow the sample type code. The location code consists of a two- to five-digit numeric or alpha-numeric code indicating the sample location. Field blanks and trip blanks will use a consecutive numbering system starting at 01, assigned in the field.

4.3 SAMPLING ROUND CODE/DUPLICATE CODE

A two-digit numerical code will be used to designate additional location information. For soil samples, the sampling code will represent the depth of the

sample in feet below the ground surface. Duplicate samples will be designated by the sampling code preceded by a 9.

Matrix spike and matrix spike duplicate samples are collected as additional sample volume at selected locations. Although identified as such on chain-of-custody records, specific sampling codes will not be provided for matrix spikes or matrix spike duplicates (MS/MSD) samples.

4.4 EXAMPLES OF SAMPLE NUMBERS

Examples of sample number codes are as follows:

- BC-SSMW03A-25 = Beloit Corporation, split-spoon soil sample from monitoring well MW03A at a depth of 25 ft.
- BC-GWMW03A-01 = Beloit Corporation, groundwater sample from monitoring well MW03A, first sampling round.
- BC-GWMW03A-91 = Beloit Corporation, duplicate groundwater sample from monitoring well MW03A, first sampling round.
- BC-SUSB10-00 = Beloit Corporation, surface soil sample from location of shallow soil boring number 10, sample collected at the ground surface.
- BC-GDDB01-30 = Beloit Corporation, groundwater sample collected while drilling deep boring DB1 at 30 ft from the ground surface.
- BC-GIIB01-50 = Beloit Corporation, groundwater sample collected while drilling intermediate boring IB1 at 50 ft from ground surface.

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GENERAL SAMPLING EQUIPMENT AND PROCEDURES

5.1 SOIL GAS SURVEY

5.1.1 Objective

The objective of the soil gas survey is to locate potential sources of VOCs at the site and identify potential near surface contaminant sources or soil gas associated with contamination at depth.

5.1.2 Personnel and Responsibilities

A team of two individuals will perform the soil gas survey. A technician will drive the probe into the ground and extract a sample into a glass vial. A site chemist will analyze the collected samples by operating the in-field gas chromatograph (GC).

5.1.3 Methods

Soil gas sample locations will be approximated in the field by using a compass to measure a bearing between soil gas points and measuring the distance between the reference points (trace). Permanent site features may be used as reference points. A detailed log will be maintained identifying reference points and associated bearing and trace. In addition, each soil gas location will be plotted on a field map as each location is identified in the field during sample acquisition activities. Additional soil gas samples may be collected at unspecified locations based on field observations and/or GC results.

Soil gas samples will be collected by:

- Driving a 1/2-in. or 5/8-in, which ever is appropriate, diameter solid rod to a depth of approximately 3 to 4 ft below ground surface to create a pilot hole.
- The rod is replaced with an AMS Retract-a-Tip gas vapor probe. This probe will be driven down the pilot hole using a slam bar.
- The screen casing is pulled back to expose the screen.

- The teflon vapor tube, which is exposed at the surface, is slipped over the sample tip of the Thermo Environmental (TE) 580B PID.
- The sample vessel (Tedlar bag) is connected to the exhaust (sample) port of the TE 580B PID with teflon tubing.
- Both ports on the Tedlar bag are opened.
- The pump (TE 580B PID) is switched on and allowed to draw in-situ soil gas through the system for a minimum of 2 minutes.
- After 2 minutes the sample port on the Tedlar bag will be closed and the bag allowed to fill.
- With the pump still running the input port of the Tedlar bag will be closed, then the pump will be switched off.
- The Tedlar bag will be disconnected from the pump, shielded from sunlight and placed on ice in a cooler.
- The pump (TE 580B PID) will be disconnected from the AMS soil gas probe teflon vapor tube.
- The screen casing will be closed (screen covered) and then the probe will be removed from the pilot hole.
- Prior to the next sampling the AMS gas vapor probe will be decontaminated as-described below, then the probe, pump and teflon tubing will be connected and purged with atmospheric air by running the pump for a minimum of 1 minute. Tedlar bags that have been used may be re-used if:
 - G.C. results show no detectable VOCs, and
 - The Tedlar bag has been decontaminated with high purity helium gas.

The external surface of the gas probe and the pilot rod will be decontaminated by wiping both with paper towels moistened with TSP or liquinox cleaning solution followed by paper towels moistened with rinse water.

- Move to next sample location.

The sample will be delivered to the in-field GC for analysis of VOCs. Results of the soil gas analyses will be plotted in the field on the aforementioned site map.

Used paper towels will be disposed in trash bags and placed with other investigative wastes. Field blanks will be collected at a frequency of one blank per ten samples, consistent with sampling of other media.

5.2 SHALLOW SOIL BORINGS AND SOIL SAMPLING

5.2.1 Objective

The objective of this activity is to chemically characterize site surface and subsurface soils, characterize potential source areas, and supplement information obtained during the soil gas survey.

5.2.2 Personnel and Responsibilities

A drill crew of two individuals will perform the shallow soil borings. A geologist will supervise their efforts, record soil boring logs, collect soil samples, and function as the Site Safety Officer.

5.2.3 Methods

Twelve shallow soil borings will be drilled to a minimum depth of 25 to 35 ft (water table) using 4 1/4-in. I.D. or 6 1/4-in. I.D. hollow stem augers. Soil samples will be collected at 2-ft intervals with a 3-in. split spoon sampler, if possible. If the 3-in. split spoon sampler will not retain the sample, a 2-in. split spoon sampler will be employed. The samples will then be visually classified in the field by a geologist, placed into appropriate sampling jars as specified in Section 3.1.2 of this Field Sampling Plan, and screened using the field headspace method. A log of the soil boring, identifying materials encountered, will be maintained by the site geologist/engineer. Soil samples will be visually classified in accordance with the Unified Soil Classification System (USCS).

Twelve surface soil samples will be collected, one at each boring location. A background surface soil sample will be collected at the location of the background monitoring wells. Surface and selected subsurface soils collected during shallow soil borings will be analyzed for U.S. EPA CLP TCL and TAL parameters.

As indicated in Section 3.1.2, subsurface soil samples will be selected for analysis based on elevated PID/FID readings, visual observations of contamination, and changes in soil stratigraphy. Selection of subsurface soil samples for analysis will be determined in the field and based on field observations at the time of sampling.

Subsurface soil samples will be evaluated for VOCs using the field headspace method. An 8 oz. glass sample jar is half filled with representative soil collected

from a 3-in. split spoon sample. A piece of aluminum foil, sufficient in size to cover the opening and threads of the sample jar, is then placed over the jar opening. The teflon lid of the jar is then secured snugly over the aluminum foil. The contents of the jar are to be heated for 30 minutes. After 30 minutes the lid is removed (keep the aluminum foil cover in place) and an 11.7 eV PID (Appendix A - A4) or FID (Appendix A - A6) probe is forced through the aluminum foil seal to collect a headspace sample from the jar. A decision to analyze samples from each location will be made after all samples at that location have been collected, screened, and visually described.

A minimum of 24 sets of subsurface soil samples with at least one collected from each boring will be selected for U.S. EPA CLP analysis. Information regarding QA/QC sample numbers and frequency are contained in Table 1-1 of this QAPP. Appropriate sample containers, preservation and packaging information are described in Table 1-2 of this QAPP and Section 3.1.2 of the FSP.

A temporary decontamination facility will be constructed on-site on Beloit Corporation property. Decontamination will include steam cleaning or high pressure hot water washing the drilling equipment and tools between boreholes, and detergent washing and water rinsing the split spoon samplers after each collected sample. Decontamination procedures are detailed in the Health and Safety Plan (Volume II of the Planning Documents).

Shallow soil borings will be abandoned in accordance with IEPAs Monitor Well Plugging and Abandonment Guidance Document (IEPA 1990) (Section 5.4.3.2 of the FSP). The disposition of excess borehole cuttings generated during drilling is discussed in Section 3.2.1.3 of the FSP.

5.3 ELECTROMAGNETIC(EM)/MAGNETOMETER SURVEY

5.3.1 Objective

The objective of the EM/magnetometer survey is to determine whether buried metal containers or underground metal tanks exist in the area of the 1314 Watts Avenue and the Soterion property in the Blackhawk Acres Subdivision.

5.3.2 Personnel and Responsibilities

A team of two individuals, including a geologist/geophysicist and an assistant will be responsible for performing these surveys. The geologist/geophysicist will operate the instrument and the assistant will help lay out grid lines, flag locations and record activities.

5.3.3 Methods

An electromagnetic conductivity meter (see Appendix A - A1) and/or a proton precession magnetometer (see Appendix A - A3) will be used to perform these surveys. The team will set up the geophysical survey grid with 10 ft spacings across each area to be surveyed. The geologist/geophysicist will traverse the area while operating the EM or magnetometer. Areas of anomalous readings will be plotted in the field with pin flags and on a site map. A base location, away from the potentially contaminated area, will be set up as a calibration verification point for the geophysical instrument. The instrument will be checked at this location before, after, and during the investigation. Instrument readings and drift at the base location will be recorded.

Intrusive activities will not be performed during these surface geophysical surveys, therefore decontamination and waste disposal procedures are not anticipated.

5.4 HYDROGEOLOGIC INVESTIGATION

5.4.1 Objective

The objective of the hydrogeologic investigation is to obtain information for characterization of the hydrogeologic setting, determine groundwater flow direction, assess the potential for preferential groundwater flow, and identify the physical characteristics of the geologic media at the site.

5.4.2 Personnel and Responsibilities

Deep and intermediate groundwater quality borings, monitoring well borings, and a geotechnical boring will be performed and installed by a drill crew of two individuals. Drilling operations will be supervised by a geologist/engineer, who will collect groundwater and soil samples, obtain natural gamma logs, and function as the Site Safety Officer. Water levels will be measured by a technician. Hydraulic conductivity tests will be performed by a geologist or field technician.

5.4.3 Methods

5.4.3.1 Deep Borings and Groundwater Sampling - Five deep (DB1-DB4 and W22C) and two intermediate (IB1 and W23B) groundwater quality borings, and one deep geotechnical boring (GB1) will be performed at locations inside the RI/FS site boundaries. The primary objective of the deep and intermediate borings is to assess groundwater quality with depth in the aquifer. The groundwater analytical data will be used exclusively as screening data to identify target VOCs in groundwater at different zones in the aquifer. The primary objective of the geotechnical boring is to provide a borehole for ground-truthing the natural gamma ray logger and for obtaining characteristic soil descriptions for the site. The proposed locations for the borings are shown on Drawing 15268-7.

Dual-tube reverse circulation drilling methods (Appendix A - A2) and plugged, screened hollow stem auger drilling methods will be used to advance the deep and intermediate boreholes, respectively. Representative groundwater samples will be collected during drilling with a stainless steel bailer or submersible pump with inflatable packer at selected zones in the aquifer. Groundwater samples will be collected every 10 ft beginning at the water table. If groundwater does not recover, or is not expected to recover within 30 minutes due to the presence of a fine grained, low permeability zone, no sample will be collected from that zone.

Groundwater samples will be collected through the center of the drill string or casing. A bailer, B-K pump, or submersible pump with an inflatable packer will be used to purge three times the standing water volume in the drill rod or auger before a sample is collected. If a packer is used, then three times the volume of the packed-off zone will be purged. Groundwater samples collected during each boring will be screened for VOCs with an in-field GC using the methods specified in the QAPP. The deep borings will be drilled to a minimum depth of 100 ft and the intermediate borings to a minimum depth of 70 ft at the proposed locations shown on Drawing 15268-7. Total actual depth is to be determined in the field based on stratigraphy and groundwater sample screening results. Drilling and groundwater sampling will be discontinued at a boring when one of the following situations develops:

- When groundwater screening results indicate that VOCs are not present and the 100 ft (for deep borings) or 70 ft (for intermediate borings) minimum depth has been reached, sampled, and analyzed;
- When groundwater screening results indicate that VOCs are present and the minimum depth requirement of 100 ft (deep borings) or 70 ft (intermediate borings) has been achieved, and a minimum of two consecutive clean groundwater samples have been collected below the deepest VOC detection in that boring; or
- For intermediate borings at potential source areas, and deep borings where non-aqueous phase has been identified, when a confining unit greater than or equal to 3 ft in thickness is encountered, irrespective of depth. In this situation, the last groundwater screening sample will be collected at the top of the confining layer.

GC results of groundwater samples will be used to assess the vertical extent of VOC contamination at each boring location, and will be used to determine installation depths for monitoring wells that may be installed during this phase of work and placement of monitoring wells that may be installed during a Phase 2 investigation.

Due to the drilling methods (dual tube and plugged hollow stem auger), split-spoon samples will not be obtained during drilling. A boring log based on discharged drill cuttings will be kept by the geologist/engineer supervising drilling activities. To supplement subsurface information, a natural gamma ray log will be obtained through the drill casing upon completion of the borehole. A Mount Sopris Model 1000-C borehole logger (Appendix A - A5) and natural gamma ray logging tool will be used to obtain the downhole geophysical log.

Geotechnical boring GB1 will be advanced to a minimum depth of 100 ft using a pure bentonite mud and rotary wash drilling methods (minimum borehole size of 4-in.). Standard penetration test (ASTM D1586) soil samples will be collected every 5 ft from 2 ft to 30 ft, every 2 ft from 30 ft to 80 ft, and every 5 ft from 80 ft to 100 ft. Representative soils will be placed in 8 oz sample jars and retained. A detailed soil borehole log will be maintained by the field geologist/engineer supervising drilling activities.

When the mud rotary drilling technique has reached a depth of 100 ft, in geotechnical boring GB1, and is able to maintain an open hole to this depth, the drill string can be removed and the borehole logged with the natural gamma ray logging device. The natural gamma ray detecting probe is lowered to the bottom of the borehole and is then advanced to the surface at a logging rate of slightly less than 1 ft/sec. This gamma ray trace will be the gamma ray tool ground-truth log for the site.

Deep and intermediate groundwater quality boreholes, not instrumented with a monitoring well, and the geotechnical boring will be abandoned in accordance with IEPAs Monitor Well Plugging and Abandonment Guidance document (IEPA, 1990). Drill cuttings and fluid will be screened with a PID during drilling operations. The disposition of excess borehole cuttings and drilling fluids generated during drilling is discussed in Section 3.2.1.3 of the FSP.

To minimize potential inadvertent contamination by drilling equipment and/or inter-borehole contamination, steam cleaning will be used between boring locations (except between borings at a well nest). The drill rig and drilling tools will be steamed cleaned before mobilization onto the site, and will be steam cleaned between each boring (except between borings at a well nest). The disposition of decon water is discussed in Section 6 of the FSP.

5.4.3.2 Monitoring Well Installation/Abandonment - Twelve groundwater monitoring wells (eight new and four replacements) are proposed including nine wells at four locations on Beloit Corporation property, one well at one location upgradient of the site, and two wells at one location downgradient of the site.

Four replacement wells are proposed for existing monitoring wells W3, W5, W8, and W11. Proposed locations are shown on Drawing 15268-7.

Existing non-functional wells and standpipes will be abandoned in accordance with IEPA monitoring well abandonment procedures (IEPA, 1990). Surface casing will be removed a minimum of 12 inches below ground surface. A neat cement slurry (5 gal water per 94 lb bag cement) will be mixed and slowly pumped into the well casing through a tremie pipe. The tremie pipe will be placed at the bottom of the well and then slowly withdrawn as the cement slurry is pumped into the well casing. Pumping will continue until the cement slurry flows out the top of the well casing. The 12 inch deep hole will then be filled with natural soils.

Water table well borings (anticipated depth: 25 to 35 ft) will be drilled using 4 1/4-in. or 6 1/4-in. I.D. hollow stem augers. If sand blow-in through the augers occurs during drilling, rotary wash methods using clear water will be used to remove material from the interior of the augers. Split spoon soil samples will be collected at 2.5 ft-intervals to the bottom of the boring (only the deep boring at a well nest will be split spoon sampled) where feasible. If sand blow-in is severe, no sampling will be conducted and natural gamma ray logging will be substituted.

Intermediate depth (anticipated depth: 60 to 70 ft) piezometer borings will be drilled using 4 1/4-in. I.D. or 6 1/4-in. I.D. hollow stem augers or rotary wash drilling methods. Split spoon soil sampling intervals (2.5 ft) will be similar to water table well borings.

Based on past subsurface drilling at the site, augering methods may not be possible below those depths where extensive sand blow-in occurs. Due to limitations in augering methods, rotary drilling methods using casing will be used when appropriate. Alternative methods include use of dual-tube reverse air rotary or clear water wash rotary. In the event clear water does not remove cuttings or the borehole is unstable, a pure bentonite mud wash may be necessary to complete the boreholes. During drilling operations, attempts will be made to minimize the amount and use of bentonite drilling mud. Bentonite with chemical additives will not be used at the site.

Water used during drilling activities will be obtained from the City of Rockton municipal water supply. As a quality assurance procedure, a sample of the water used for drilling and other activities at the site will be collected and analyzed for VOCs (CLP SOW OLMO2.0) during initial drilling activities.

A log of the monitoring well boring will be recorded by the site geologist. Soil samples will be visually classified in accordance with the Unified Soil

Classification System (USCS). Twenty (20) soil samples will be analyzed by a soils laboratory for grain size. Samples will be selected following completion of the drilling program to obtain a representative selection of the various soil types encountered during the investigation. The purpose of these analyses is to confirm field identification of soils and to assist in assessing subsurface soils as a contaminant migration pathway. Soil samples will be field screened in the open atmosphere with a PID to assess potential hazards to the field personnel. The disposition of excess soils is discussed in Section 3.2.1.3₂ of the FSP.

Final soil boring logs will be prepared based on field observations, natural gamma ray log response, and soil testing and laboratory sample classification. Soil samples will be retained for Beloit Corporation until termination of the RI/FS. Procedures for sample handling are discussed in the QAPP.

Monitoring wells will be constructed as shown in Drawings 15268-A7 and 15268-A8 in the Work Plan. Water table wells will be constructed with 10-ft long, No. 10 slot (0.010-in.) continuous wrap 2-in. I.D. stainless steel screens placed so the screen is likely to intercept the water table. Piezometers will be constructed with 5-ft long No. 10 slot (0.010-in.) continuous wrap 2-in. I.D. stainless steel screens and a 10 ft section of stainless steel riser. The riser above the 10 ft stainless steel riser section will consist of 2-in. I.D. schedule 40 PVG pipe for wells less than 80 ft deep and schedule 80 PVC pipe for wells greater than 80 ft deep.

The annular space between the well and the edge of the borehole will be backfilled with clean silica sand to approximately 2 ft above the top of the well screen. A minimum 3-ft hydrated bentonite pellet seal will be placed directly above the sand pack. At water table wells, the annulus will be backfilled with granular bentonite. At piezometers, a fine silica sand or bentonite pellets will be placed above the sand filter pack and bentonite slurry placed above the fine silica sand or bentonite pellets, using a tremie pipe. A granular bentonite surface seal will be installed at each well. A locking steel protective casing will be installed at the surface of each well.

The following partial abandonment procedures are to be followed when installing monitoring wells in water quality borings.

If the backfill interval is greater than 50 ft then the water quality borehole is to be abandoned following IEPA (1990) monitoring well abandonment guidelines (see paragraph 2, Section 5.4.3.2). A monitoring well will then be installed in a new borehole that will be drilled approximately 5 ft from the abandoned water quality borehole.

If the backfill interval is less than 50 ft then the following guidelines will be used to install monitoring wells in water quality borings:

- A tremie pipe will be used to install approximately 3/8-in. diameter bentonite pellets or chips (interlayered with a clean silica sand in permeable zones) in water quality borings that are less than 250 ft deep and with a standing water depth of less than 150 ft.
- 6.25-in. I.D. hollow-stem augers will be used to advance intermediate water quality boreholes that may require partial abandonment.

Bentonite material must be installed at the base (minimum 2 ft thick) and top (minimum 2 ft thick) of the backfill interval and must also extend through impermeable zones. In the permeable zones a minimum of 2 ft of bentonite material will be installed for every 4 ft of silica sand. If the backfill interval is 10 ft or less then bentonite will be the only backfill material utilized.

New monitoring wells will be developed by alternatively surging and purging the wells for a minimum of 30 min. using a bailer. After the surge and purge cycles are completed, the well will be pumped until 10 well volumes are removed or until the pH and specific conductance of water from the well has stabilized. The disposition of purge water is discussed in Section 3.2.1.3 of the FSP.

To minimize potential inadvertent contamination by drilling and sampling equipment, and/or inter-borehole contamination, steam cleaning will be used between borings (except between borings at a well nest). The drill rig and drilling tools will be steamed cleaned before mobilization onto the site, and will be steam cleaned between each boring. The split spoon sampler will be cleaned in a liquinox or non-phosphate detergent solution between monitoring well boreholes except at well nests. The disposition of decon water is discussed in detail in Section 6 of the FSP. PVC and stainless steel well pipe and stainless steel screen will be steam cleaned or washed with a high pressure hot water washer prior to installation.

5.4.3.3 In Situ Hydraulic Conductivity Tests - Hydraulic conductivity will be measured by baildown testing in water table wells and/or by air pressure methods in deeper piezometers. The Bouwer and Rice Method (1976) will be used to analyze the baildown test data. The methods to be used are as follows:

- Measure water level with a tape and sounding device or electronic water level indicator.

- Place the pressure transducer into the well and allow approximately three minutes for the probe to equilibrate to the water temperature and pressure.
- Install the well head pressure device to seal the well head (for piezometers only).
- Enter the reference water level into the data logger and check the water level using the pressure transducer until water level reading is stable.
- After a stabilized water level reading is obtained from the pressure transducer, the well (piezometers only) is pressurized with sufficient air pressure to displace several ft of water (0.4 PSI/ft of water).
- Air pressure is maintained until the water level reading from the transducer is constant (for piezometers only).
- The air pressure is then instantaneously released while running the pressure transducer recorder in the log sampling mode (for piezometers only).
- At water table wells a single bailer of groundwater is removed to reduce the water level, while recording the pressure transducer readings with the data logger.
- The test results may be immediately printed out to obtain a hard copy.
- Data are transferred from the data logger to a computer for analysis.

5.4.3.4 Water Level Measurements - Water levels in existing and new monitoring wells will be measured. An electric water level indicator will be lowered into the monitoring wells until the water level is reached. Depths will be recorded to the nearest 0.01 ft.

5.5 SURFACE WATER AND SEDIMENT INVESTIGATION

5.5.1 Objective

The objective of this activity is to determine surface water drainage patterns at and around the site. The staff gauge measurements will be used in the general assessment of site water levels and the affects of surface water/groundwater flow interactions.

5.5.2 Personnel and Responsibilities

A survey crew or field technician will install three staff gauges in the Rock River. A technician will measure water levels. A scientist or engineer will identify and map drainage patterns.

5.5.3 Methods

Staff gauges will be metal rods with a scale in hundreds of feet. The gauge will be installed at least one foot into the river sediments, or until stable, so the scale extends at least one foot above and below the water line. The reference line on each gauge will be surveyed for location and elevation. Surface water level measurements will be obtained at the staff gauge locations concurrently with groundwater level measurements. Measurements will be made by reading the water level mark on the staff gauge and subtracting the measurement from the staff gauge reference elevation.

A scientist or engineer will visually locate, identify, and map drainage patterns at the site. The drainage patterns will be plotted on a drawing developed from the site base map. The data will be used to assess the potential for erosional transport of contaminants off-site.

5.6 SURFACE SOILS ASSESSMENT

5.6.1 Objective

The objective of this activity is to assess the potential for direct contact exposure to contaminated surface soils.

5.6.2 Personnel and Responsibilities

A geologist will collect surface soil samples at the twelve shallow boring locations and the background location. The background location is anticipated to coincide with the background monitoring well location. A scientist or engineer will identify the erosion potential of surface soils for assessing the surface soil contamination pathway.

5.6.3 Methods

An inspection of the site will be conducted. Surface soils will be observed and monitored with a PID during sampling activities. Based on this site inspection and analytical data collected during Source Characterization and Migration Pathway Assessment activities, the need for additional surface soil sampling will be determined.

5.7 AIR ASSESSMENT

5.7.1 Objective

The objective of this activity is to assess the potential for release of contaminants to air.

5.7.2 Personnel and Responsibilities

The geologist/engineer on each drilling rig will monitor air quality using a PID during soil borings and monitoring well installation.

5.7.3 Methods

Air quality will be monitored using a PID. Monitoring results above background will be recorded in each individuals field logbook. Monitoring during intrusive activities is assumed to approximate "worst case conditions", when potentially contaminated soil or waste is exposed to the atmosphere.

5.8 GROUNDWATER SAMPLING

5.8.1 Objectives

The objective of groundwater sampling is to determine the nature, magnitude and extent of groundwater contamination.

5.8.2 Personnel and Responsibilities

A crew of two technicians will collect samples.

5.8.3 Methods

Groundwater sampling will proceed from wells expected to have the lowest contaminant concentrations (based upon observations during drilling and existing groundwater quality data), to the wells suspected of having the highest contaminant concentrations. Each sampled well will be purged immediately prior to sampling using a submersible sampling pump at a high pumping rate. A packer will be used in all wells screened below the water table, and inflated within the stainless steel riser so as to seal off the PVC riser.

The volume of water removed from the well will be measured so that a minimum of three volumes are removed. Specific conductance, temperature and pH will also be monitored for stabilization during purging. After well purging has been completed, the pumping rate will be decreased to approximately 100 ml/min and the VOC sample collected directly from the pump discharge. Sample bottles, filtering, and preservation will be as specified in Table 1-2 of the QAPP.

The disposition of purged water is discussed in Section 3.2.1.3 of the FSP.

The sampling equipment and water level measurement tape will be cleaned between wells with a Liquinox or another non-phosphate detergent solution and rinsed with deionized water. Decontamination of the pump and tubing will consist of pumping the decon solution into the wash tanks for several minutes. The pump will then be moved into the rinse tank and pumping will resume with rinse water.

Groundwater sample blanks will be collected by pumping deionized water through the sampling pump into the sample bottles. Matrix spike/matrix spike duplicate samples will be collected using the same sampling device. Duplicate samples for volatile organics analysis will be obtained by alternately filling VOC vials from the pump. The remainder of the sample will be split between the principal sample and the duplicate sample.

[mad-603-15g]

DECONTAMINATION PROCEDURES

Procedures to be followed to decontaminate equipment and personnel are described in the Site Health and Safety Plan. The procedures are summarized below.

Site Personnel Decontamination Procedure:

- Equipment drop;
- Wash outer booties and rinse;
- Dispose of outer latex booties;
- Wash boots in Liquinox bootwash;
- Clean outer gloves in Liquinox wash solution (discard if too soiled to clean thoroughly);
- Dispose of polycoated Tyvek suits;
- Dispose of surgical gloves;
- Wash hands in hand wash;
- Wash face and neck in face wash; and
- Clean and sanitize face mask.

Site personnel will perform the above mentioned decontamination procedure prior to leaving the site. Discarded clothing and other articles will be collected in double-lined, heavy duty garbage bags.

Equipment and vehicle decontamination procedure:

- Decontamination will be performed prior to site entry;
- Decontamination will be performed on site;
- Gross contamination will be removed with a brush and Liquinox solution; and
- Steam cleaning or hot water high pressure washing will follow.

The drilling equipment will be steam cleaned or high pressure hot water washed at the temporary decon facility and between boring locations prior to exiting the site. Another area, located away from the equipment decontamination area, will be designated as a site personnel decontamination area.

Decontamination will include steam cleaning or hot water high pressure washing the drill rig, drilling equipment, and tools between boreholes, and detergent washing and water rinsing the split spoon samplers after each collected sample. The drill rig and equipment may also require scrubbing of accessible parts with a detergent/water solution. Well materials will also be steam cleaned or hot water high pressure washed and wrapped in plastic until installed. The bailer cable, trowels, spatulas, stainless steel bucket and water level measurement tape will be cleaned with Liquinox or another non-phosphate detergent solution, and rinsed with deionized water.

All decontamination water will be captured by the decontamination pad, pumped into appropriate storage containers and then routed to the local POTW for treatment, assuming that approval can be obtained from the POTW. If approval can not be obtained, these materials will also be contained, stored in a secure area on Beloit Corporation property and disposed of at the time of the final remedy.

[mad-603-15h]

SAMPLE HANDLING AND ANALYSIS

7.1 PARAMETERS

Samples collected for chemical analysis will be analyzed by a laboratory approved by the U.S. EPA Region V Contract Program Management Section (CPMS). Chemical parameters for which groundwater will be analyzed are:

- U.S. EPA CLP TCL organics;
- U.S. EPA CLP TAL inorganics;
- Field pH;
- Field-specific conductance;
- Chloride;
- Sulfate;
- Alkalinity;
- Total phenolics;
- Nitrate-Nitrite nitrogen.
- Ammonia nitrogen; and
- Total dissolved solids.

Measurement of pH, specific conductance, and temperature will be performed in the field. The groundwater samples to be analyzed for U.S. EPA CLP TAL metals will be field filtered with 0.45 micron filters, as soon as possible after collection. Shallow soil boring samples (soil) will be analyzed for U.S. EPA TCL and TAL parameters. Select samples from monitoring well installation borings will be analyzed for grain size by a soils laboratory.

7.2 SAMPLE PRESERVATION

Samples will be collected and preserved in a manner appropriate for the analyses they receive (see Table 1-2 of the QAPP). The portion of groundwater samples requiring field filtering prior to analysis (see Table 1-1 of the QAPP) will be filtered using a pressure filtration device, through a 0.45 micron filter, as soon as possible after collection. Filtered portions of the samples will be preserved, as appropriate, immediately after filtration. Sample fractions will be preserved before shipment according to the procedures shown in Table 1-2 of the QAPP.

Preservatives added to the samples will be prepared using reagent grade chemicals. Table 1-2 of the QAPP should be consulted for details regarding sample packaging and shipping.

[mad-603-15i]

SAMPLE DOCUMENTATION

Field sampling activities will be documented using a bound waterproof notebook/logbook. Information recorded in the field logbook will include date of sampling, sampler, weather conditions, observations, and methods of preservation. Additional data pertaining to sampling may also be included in the logbook.

Samples will be collected under chain-of-custody procedures. Standard forms including sample labels, sample tags, chain-of-custody forms, and custody seals used for sample tracking will be maintained (see attachments). A brief description of sample documents follow:

A. Chain of Custody Form

1. One form per shipping container (cooler).
2. Carrier service does not need to sign form, if custody seals remain intact.
3. Use for all samples.

B. Chain-of-Custody Seals

1. Two seals per shipping container to secure the lid and provide evidence that samples have not been tampered with.
2. Cover seals with clear tape.
3. Record seal numbers on Chain-of-Custody Form.
4. Use for all samples.

C. Sample Tags

1. Each sample container must have a sample tag affixed to it.
2. Sample tag numbers are recorded on the Chain-of-Custody Forms.

3. Use for all samples.

D. Sample Identification Record Form will:

1. Provide means of recording crucial sample shipping and tracking information.
2. Contain information such as:
 - Sample number
 - Sample matrix
 - Sample location code
 - Sample round
 - Chain-of-custody number
 - Lab code
 - Date sampled
 - Date shipped
 - Airbill number
 - Sampling tag number

Paperwork accompanying the samples being shipped to the laboratory will be sealed in a plastic bag that is taped to the inside of the cooler lid. Copies of the chain-of-custody forms, and other paperwork (if possible), will be retained with the field files.

Two sample seals will be placed on opposite sides of the lid and extending down the sides of the cooler. The lid will be securely taped shut prior to shipment.

[mad-603-15j]

Tables

TABLE 1

SUMMARY OF DATA GENERATING ACTIVITIES
AND ASSOCIATED QUALITY OBJECTIVES
BELOIT CORPORATION ROCKTON FACILITY
ROCKTON, ILLINOIS

Activity	Use of Data	Data Quality Objective
TASK 1		
• Site Mapping	Prepare a site base map for use in developing other maps showing RI/FS data.	Accurately depict existing conditions, and prepare accurate RI data interpretation presentations.
• Evaluation of Existing Monitoring Wells	Assess the condition of existing wells at the site. Supplement the evaluation of geologic conditions at the site.	Identification of wells to be used during the RI.
TASK 2		
SOURCE CHARACTERIZATION		
• Electromagnetic/Magnetometer Survey	Assess the presence and location of buried metal drums and/or underground storage tanks.	Obtain consistent relative differences among sampling points.
• Soil Gas Survey	Identify potential contaminant source areas. To optimize placement of groundwater quality and soil borings.	Identification of VOCs present in soil gas samples. Obtain relative differences among sampling points.
• Shallow Soil Borings and Soil Sampling (surface and subsurface soils)	Collect surface and subsurface soils and quantify contamination in potential source areas.	Identification of compounds present at source areas. Meet performance criteria for TCL parameters for organics and TAL parameters for inorganics as stated in the QAPP.

TABLE 1
(continued)

Activity	Use of Data	Data Quality Objective
MIGRATION PATHWAY ASSESSMENT		
Hydrogeologic Investigation		
• Deep and Intermediate Groundwater Quality Borings	Define hydrostratigraphy, subsurface geology and the potential for preferential groundwater flow pathways.	Screen the vertical extent of contaminant migration. Meet the performance criteria for methods described in the FSP.
• Monitoring Well Installation and Replacement	Characterize site geology and hydrogeology. Obtain groundwater samples for assessment of contaminant migration pathways.	Meet performance criteria for methods described in the FSP. Accurately and consistently describe the distribution of unconsolidated deposits.
• Hydraulic Conductivity Testing	Assess hydraulic conductivity rates at the site.	Obtain valid test results.
• Groundwater Level Measurements	Define site water table and groundwater flow directions. Evaluate vertical and horizontal hydraulic gradients; assist in evaluation of potential changes in contaminant migration.	Accurate water elevation measurements to the ± 0.01 ft.
• Location and Elevation Survey	Locate soil borings, monitoring wells and other sampling locations.	Locate sampling points to an accuracy of ± 1 ft horizontally; and monitoring wells to an accuracy of ± 0.01 ft vertically.
• Surface Water and Sediment Assessment	Evaluate drainage patterns and runoff characteristics.	Inspection of the site by a qualified scientist.
• Air Assessment	Assess air migration pathway through health and safety monitoring during site activities.	Accurate collection of data with health and safety monitoring instruments.

TABLE 1
(continued)

Activity	Use of Data	Data Quality Objective
Contaminant Characterization		
• Groundwater Sampling	Provide preliminary assessment of contaminant source impact on groundwater quality, and to aid in assessment of groundwater migration pathways.	Meet performance criteria for TCL organics as stated in CLP SOP's. Meet performance criteria for inorganics and general water quality indicator parameters stated with method descriptions in the QAPP.

TJK/km1/JEM/KJD/KJQ
[mad-403-09]

Appendix A-A

Equipment

Appendix A-A1

Operator's Manual for the EM-31D Electromagnetic Conductivity Meter



GEONICS LIMITED

1745 Meyerside Dr. Unit 8 Mississauga, Ontario Canada L5T 1C5

Tel. (416) 676-9580

Telex 06-968688

Cables: Geonics

OPERATING MANUAL

for

EM31-D

NON-CONTACTING

• TERRAIN CONDUCTIVITY METER

Revised June 1984.

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1. INTRODUCTION

Measurement of ground resistivity is one of the oldest geophysical techniques. Table 1, taken directly from Heiland*, lists typical values of resistivity for a variety of geological materials (pages 4-7). The values given are in ohm-centimeters and must be divided by one hundred to give ohm-meters.

It will be observed that in most cases the actual resistivity itself is not diagnostic and a knowledge of the way in which the resistivity varies laterally and with depth is of great importance, since this permits us to "see" features as a result of their shape rather than their actual resistivity values. There is thus a requirement for instrumentation which permits the rapid and accurate measurement of terrain resistivity. Since the EM31 does not require electrical contact with the ground it fulfills this objective.

The basic principle of operation of the EM31 is simple. With reference to Fig. 1 a transmitter coil located at one end of the instrument induces circular eddy current loops in the earth. Under certain conditions fulfilled in the design of the EM31 the magnitude of any one of these current loops is directly proportional to the terrain conductivity in the vicinity of that loop. Each one of the current loops generates a magnetic field which is proportional to the value of the current flowing within that loop. A part of the magnetic field from each loop is intercepted by the receiver coil and results

* Heiland, C.A. Geophysical Exploration. Hafner Publishing Co., New York 1968

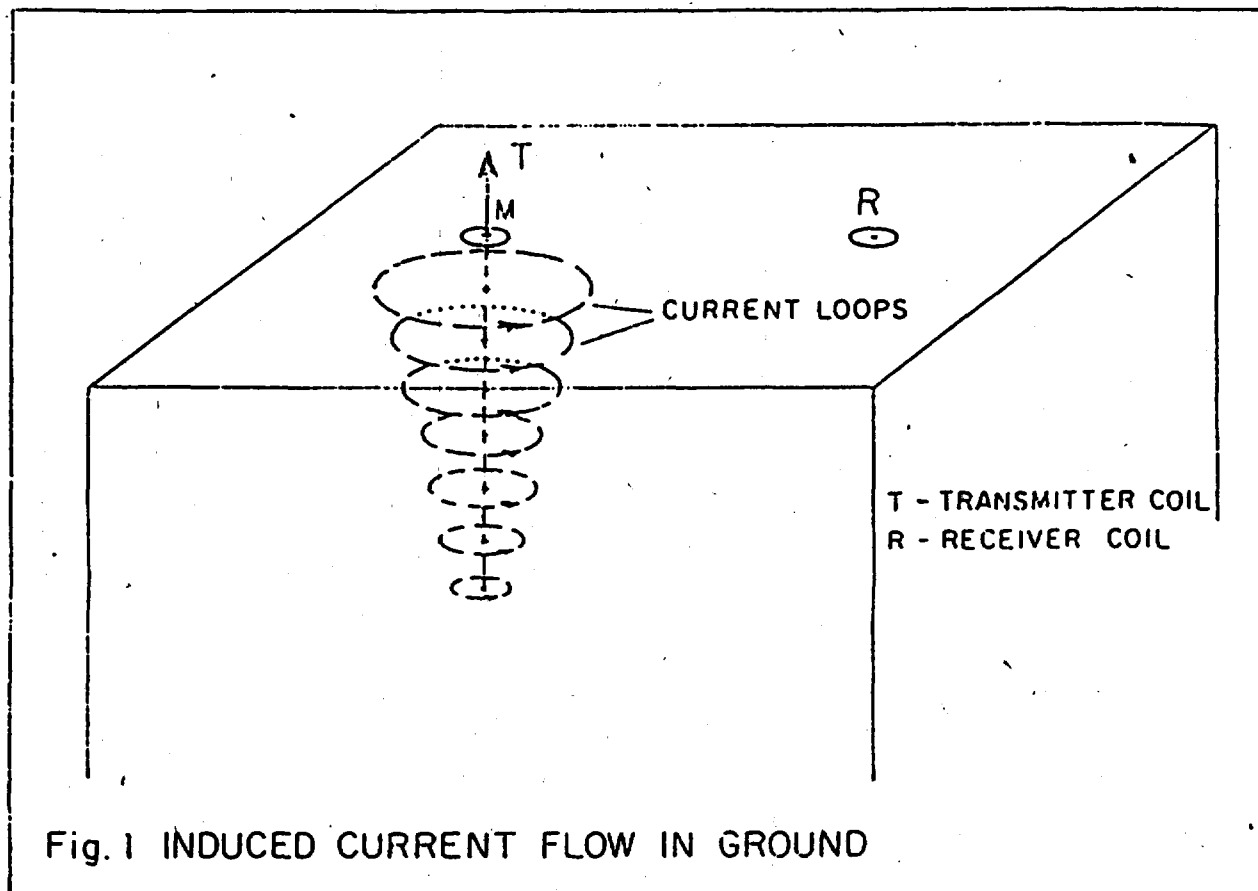


Fig. 1 INDUCED CURRENT FLOW IN GROUND

1. INTRODUCTION (cont'd)

in an output voltage which is therefore also linearly related to the terrain conductivity.

This instrument is calibrated to read the correct conductivity when the earth is uniform. In the event that the earth is layered, with each layer of different conductivity, the instrument will read an intermediate value as discussed in more detail in Section 5.2.

The unit of conductivity used is the millimho per meter. To obtain resistivity in ohm-meters the instrument reading is divided into 1000 - i.e., a reading of four millimhos per meter divided into 1000 gives two hundred and fifty ohm-meters.

Theoretical calculations show, as will be quickly evident to the operator, that the reading obtained is essentially independent of the orientation of the instrument with respect to the earth. There is, however, a small dependence on the height above the ground; lifting the instrument from the surface of a uniform earth to the normal operating height of about one meter results in a reduction in the reading of 12%. The calibration has been adjusted at the factory so that the instrument reads correctly over a uniform half-space when worn as shown in the data sheet. If the earth is layered, raising the instrument from the surface of the earth to the normal operating position can result in a reading which stays constant or even increases slightly with height. In general, readings made with the instrument at hip height will be sufficiently accurate, but for maximum accuracy the instrument can be laid on the ground as discussed in Section 5.2.

There are two components of the induced magnetic field measured by the EM31. The first is the quadrature-phase component which gives the ground conductivity measurement as described. The second is the inphase component used primarily in the EM31 for calibration purposes. The inphase component however is significantly more sensitive to large metallic objects and hence very useful when looking for buried metal drums. Although this component has been available in the standard EM31 by operating in the compensation mode (Section 2.2), both components are now available in the EM31-D from an analog output connector when coupled to a dual channel recorder.

BATTERY
PACK

OF
CONTROL

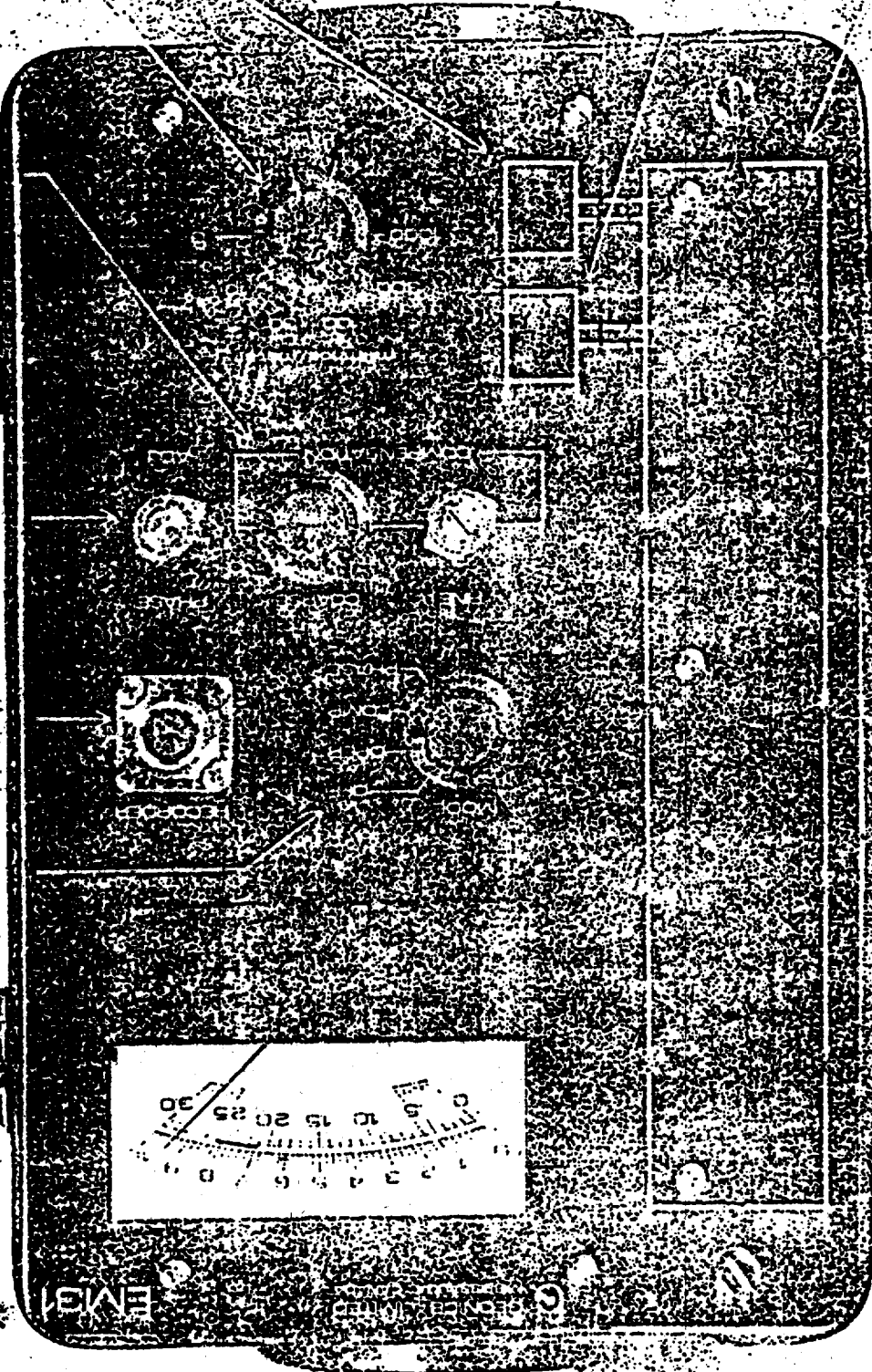
RANGE
SWITCH
DC ZERO
CONTROL

PRIMARY
FIELD
COMPEN-
SATION
CONTROL

PHASE
CONTROL

ANALOG
RECORDER
CONNECT

MODE
SELECTOR
SWITCH



EMERGENCY

EM31-D SPECIFICATIONS

Measured Quantities	(1) Apparent conductivity of the ground in millimhos per meter (2) Inphase component of the induced magnetic field
Primary Field Source	Self-contained dipole transmitter
Sensor	Self-contained dipole receiver
Intercoil Spacing	3.66 meters
Operating Frequency	9.3 kHz
Power Supply	8 disposable alkaline 'C' cells (approx. 20 hrs life continuous use)
Conductivity Ranges	3, 10, 30, 100, 300, 1000 mmhos/meter
Measurement Precision	$\pm 2\%$ of full scale
Measurement Accuracy	$\pm 5\%$ at 20 millimhos per meter
Noise Level	<0.1 millimhos per meter
Operator Controls	<ul style="list-style-type: none">• Mode Switch• Conductivity Range Switch• Phasing Potentiometer• Coarse Inphase Compensation• Fine Inphase Compensation
Analog Output	Connector on EM31-D for dual channel analog recording of ground conductivity and inphase component
Dimensions	Boom : 4.0 meters extended 1.4 meters stored Console : 24 x 20 x 18 cm Shipping Crate: 155 x 42 x 28 cm
Weight	Instrument Weight: 9 kgm Shipping Weight: 23 kgm

2. OPERATING INSTRUCTIONS

The EM31 can be used both to measure the electrical conductivity of the ground and to detect buried metal objects. Section 2.1 describes the procedure for measuring ground conductivity and section 2.2 for buried metal detection.

2.1 Ground Conductivity Measurements

2.1.1 Initial Set-up Procedure

- a) Using the identifying labels on the tubes select the transmitter coil tube, align it with respect to the main tube, insert it and fix it with the clamp.
- b) Check the battery condition, plus and minus, by setting the Mode switch (MODE SELECTOR SWITCH) to the OPER position and the Range switch to the +B and -B positions respectively. If the needle reads inside the BATT mark on the meter, batteries are in good condition, otherwise replace the batteries with a fresh set of C size alkaline batteries.
- c) Check the zero reading by setting the Mode switch to the OPER position and the Range switch to the least sensitive position 1,000 mmhos/meter (this minimizes any external noise interference while checking the zero position). If a zero adjustment is required adjust the DC ZERO CONTROL located under the front panel to obtain a zero reading. To do this the battery pack must be removed to gain access to the controls.

- d) Align and connect the receiver coil tube to the main frame tube. The instrument is now ready to proceed with the functional checks.

2.1.2 Equipment Functional Checks

The Range switch should be set at 30 millimhos/meter position for all the following tests. (If the reading is off scale, i.e., greater than 30 millimhos/meter, see note.)

- a) Set the Mode switch to the COMP position and adjust the meter reading to zero using the COARSE and FINE COMPENSATION controls.
- b) To check the phasing of the instrument set the Mode switch to the PHASE position. Note the meter reading and rotate the COARSE control one step clockwise. If the meter reading remained the same, the phasing is already correct; return the COARSE control to its original position (one step counter clockwise) and no further adjustment is necessary.

If there was a difference in the meter readings taken before and after the COARSE control was rotated one step clockwise then a phase adjustment is required. With the COARSE control in its original position adjust the PHASE potentiometer about 1/4 turn clockwise and note the new meter reading. Rotate the COARSE control one step clockwise, take a reading, and return the COARSE control to its original position. If the difference in meter readings has decreased, repeat the procedure using a further clockwise adjustment, until rotating the COARSE control the one step clockwise produces no change in the meter reading.

If, on the other hand, the difference in meter readings was increased, the PHASE potentiometer should be rotated in a counter clockwise direction instead and the procedure described above repeated until there is no change in the meter readings. Always remember to set the COARSE control back to its original position. This can be confirmed by setting the Mode switch in the COMP position and checking that the meter reads zero. If it does not read zero, repeat steps (a) and (b).

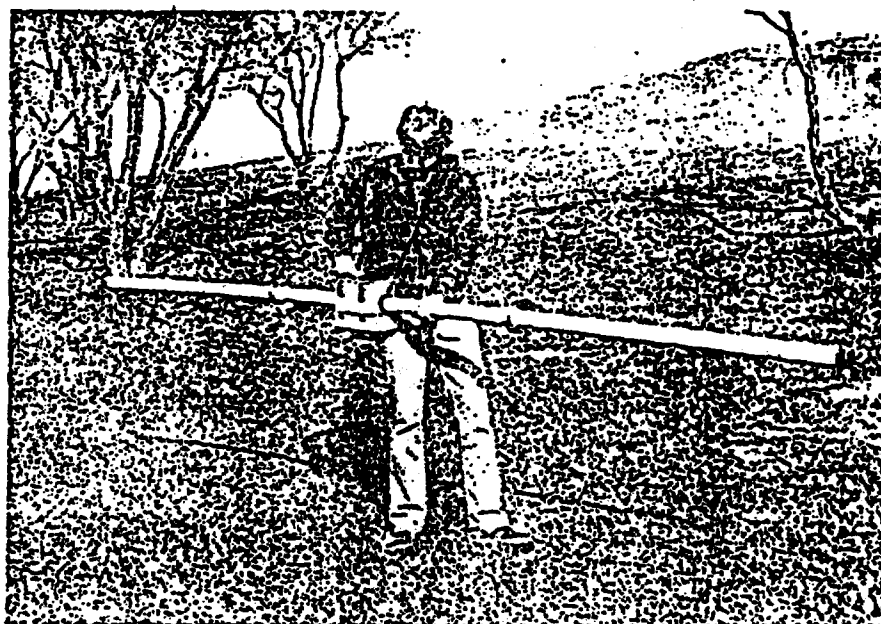
- c) To check the sensitivity of the instrument, set the Mode switch to the COMP position and rotate the COARSE control clockwise one step. The meter should read between 75% and 85% (22 to 26 millimhos/meter) of full scale deflection (inside black mark). It is unlikely that the sensitivity of the instrument will vary, however it may be useful to record the actual meter reading for comparison at a later date.

Return the COARSE switch to its original position and the EM31 is now ready to make ground conductivity measurements.

NOTE: When conducting the functional tests over ground of higher conductivity than 30 millimhos/meter, the Range switch should be set at the appropriate level. At whatever level the Range switch is in, the reading taken in (c) should still be between 22 and 26 millimhos/meter.

2.1.3 Operating Procedure

- a) Wearing the instrument with the shoulder strap adjusted so that the instrument rests comfortably on the hip as shown, switch the Mode switch to the OPER position and rotate the Range switch so that the meter reads in the upper two thirds of the scale. The full scale deflection is now indicated by the Range switch and the instrument is reading the terrain conductivity directly in millimhos per meter.



- b) The EM31 can be operated continuously while moving from one measurement station to the next however, the instrument has a time constant of about one second for which the operator should adjust his walking speed to obtain greatest accuracy.

Alternatively, to extend battery life the instrument can be switched on at each measurement station when not using a recorder. The operator will notice that the type of integrator used results in a slight initial overshoot of the needle at turn on. This is normal, and that at least two seconds should be allowed after turn on before the measurement is taken.

2.2 Buried Metal Detection

2.2.1. Set-up and Operating Procedure

The inphase component of the magnetic field is significantly more sensitive to large metallic objects than the quadrature-phase component used for ground conductivity measurements.

- a) The inphase component is readily measured with the EM31 by simply taking the reading with the Mode switch in the COMP position rather than in the OPER position.

It is recommended that the 30 mmho/m range be used since it usually gives adequate sensitivity, however more or less sensitive positions of the Range switch may also be used.

- b) To carry out a survey measuring the inphase component set the Mode switch to the COMP position and adjust the COARSE and FINE compensation controls so that a meter deflection of about 20% of full scale deflection is obtained. (The procedure of adjusting to 20% of the full scale deflection rather than to zero is only a convenience to allow for negative readings on the meter and under certain conditions a possible change in the reference level. For example, a sudden jar to the instrument can result in a small positive or negative change in the inphase reference level).

The survey is then carried out exactly as if the conductivity were being measured.

- c) This lack of a true zero reference should not cause any serious problems since when using the inphase component one is usually only looking for buried metallic objects.

These will be easily recognizable by relatively localized meter deflections occurring either singly or in a series of responses depending on the number of buried objects, their spacing and depth of burial.

Notes (1): The 20% offset should only be used when the inphase component only is being measured continuously. If both conductivity (quadrature phase) and inphase measurements are being made at each station the procedure described in 2.1.2 (a) should be used so that accurate ground conductivity measurements are obtained. If negative inphase measurements are obtained one should check the inphase reference level and/or the area for buried metallic objects or utilities.

- (2) Similarly when the dual channel recorder is being used there should be no zero offset as described in 2.2.1 (b) and the meter reading set to zero using the COARSE and FINE controls as described in 2.1.2 (a).

A zero offset for negative inphase readings can however be allowed for on the recorder by setting the zero position of the pen at some arbitrary positive value when calibrating the recorder.

3. INSTRUMENT CALIBRATION

Prior to shipping, the instrument is calibrated in the factory to read properly. If necessary, calibration procedures are easily carried out as described below.

IMPORTANT The most critical adjustment is the QF (quadrature fine) potentiometer which has been precisely adjusted at the factory.

Before any adjustments are made it is strongly recommended that the instrument first be set up at a fixed height over a known location and the ground conductivity carefully noted. If this adjustment is misaligned the instrument will have to be recalibrated over ground of known conductivity.

3.1 Null Calibration

The zero setting of the EM31 can be readily set by following the procedure described in section 2.1.1 (c).

3.2 Absolute Calibration

Absolute instrument calibration is easily achieved if an area of ground is available of known and constant conductivity down to the depth of penetration of the instrument. The procedure is simple; the instrument is located over the known area at the usual operating height (approximately 1m) and the QF compensation control is adjusted until the meter reads the correct terrain conductivity. If the ground conductivity is high the graph of Fig. 3 must be used to correctly set the instrument reading.

It is wise to maintain such an area as a calibration check area even if the variation of the conductivity with depth at that area is not accurately known. This is useful for cross checking with future measurements.

Note:

The QF and NULL controls are located under the front panel. Battery pack should be removed to have access.

4. SURVEY TECHNIQUE

Surveying with the EM31 is straightforward. As pointed out in a previous section, measurements may be made either continuously or on a station-by-station basis. In either case it is always recommended, as for any other geophysical survey, that survey lines and measurement stations be carefully laid out, and the survey performed in a systematic fashion with the resulting data accurately plotted for each measurement station. The most common survey error is to have the survey lines too short, in which case they do not extend sufficiently far off the supposedly anomalous region to permit the operator to establish the background values of terrain conductivity.

The EM31 yields good spatial resolution and measurement stations should be ten meters apart if the maximum resolution is to be employed. In many cases this spacing will be smaller than necessary, and twenty or perhaps forty meters will be adequate. The decision as to the correct spacing will be based on a knowledge of the lateral dimensions of the anticipated resistivity anomaly. To ensure the correct spacing it is useful, at the start of the survey, to continuously observe the conductivity values that are encountered as the operator moves down the survey line.

The resolution in conductivity of the EM31 is also high, with changes of 5% being quickly perceived. This instrument is capable of giving an extremely precise survey with information on small variations in the terrain conductivity.

4. SURVEY TECHNIQUE (cont'd)

It was seen in Section 1 that current flow within the earth consists of a series of concentric circles, assuming that the conductivity is laterally uniform. Therefore in the case of a uniform half-space, rotation of the instrument in a horizontal plane about the transmitter coil as a pivot will produce no change in the meter reading. Conversely, any change in the reading as this procedure is carried out is an indication of lateral inhomogeneities in conductivity. It is simpler and usually sufficiently accurate for the operator to rotate the instrument through 90° using himself as pivot at each measurement station. Thus if the lines are in a north-south direction the operator would normally walk along the line with the instrument pointing in a north-south direction; at each measurement station he can also take a reading with the instrument pointing east-west to check that this is essentially the same as the north-south reading. In the event that this reading is significantly different it may be worthwhile for the operator to then rotate the instrument to the points where the conductivity reading is both a maximum and a minimum, and to record both values. The average value can then be used for the data reduction.

The EM31 is somewhat sensitive to underground conductors such as large pipes, etc. These are usually easily recognized by the large meter fluctuations which occur within a short distance, as shown in Fig. 2. In an actual survey, since the negative-going peak is often off-scale, it is not possible to use it to locate the pipe; finding the point half way between the two positive-

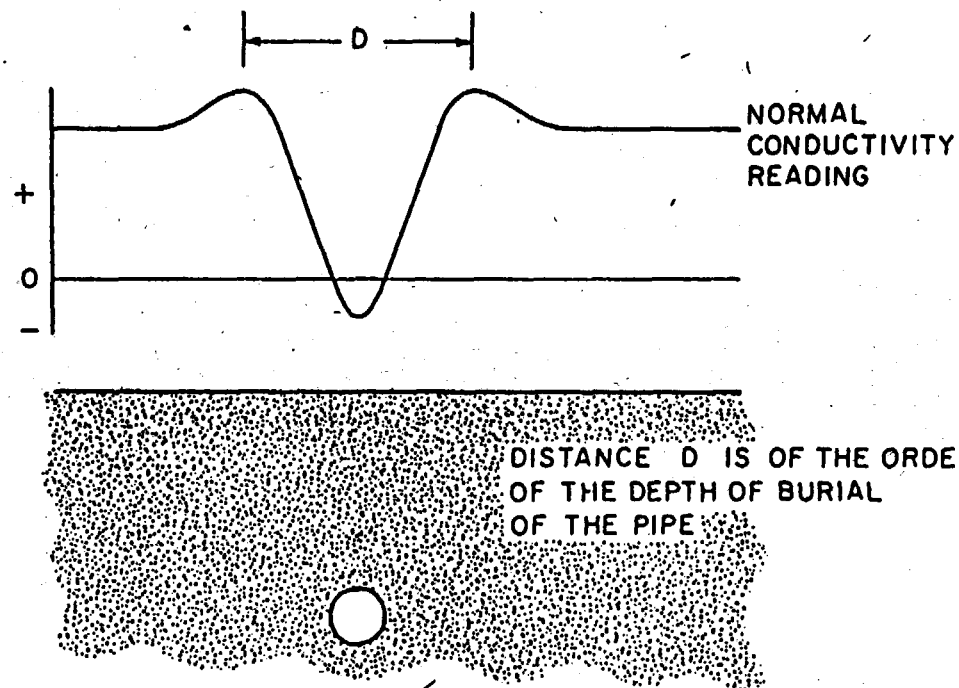


Fig.2 TYPICAL RESPONSE OVER A PIPE

4. SURVEY TECHNIQUE (cont'd)

going peaks approximately performs the same task. It is then possible to accurately determine the location and strike direction (azimuth) of the conductor axis as follows: the approximate location is determined as above, and a traverse is then made over the conductor with the EM31 pointing in the approximate direction of the conductor axis. The meter reading will now be a positive maximum when the instrument is both directly over the conductor and pointing accurately along the conductor axis.

The instrument is relatively unaffected by fences, overhead power lines, and other nearby metallic objects. In order to determine whether the reading is influenced by such structures the operator should rotate the instrument to check for changes in reading, becoming suspicious if a maximum or minimum occurs when the instrument points either perpendicular or parallel to the structure. Before recording the measurement the operator should move away from the structure until no evidence of lateral inhomogeneity is seen when the instrument is rotated.

It should be remembered that the EM31 is an electromagnetic tool and care should be taken near obvious conductors until the operator has satisfied himself as to their possible affect. In every case this is determined by rotating the instrument and determining whether there is a maximum and minimum which appears to be related to the structure. If a structure is giving such an effect it is not advisable to take the average value of the two readings as an indicator of the terrain conductivity.

4. SURVEY TECHNIQUE (cont'd)

In general the conductivity readings obtained with the EM31 will vary smoothly from one region to another. In some cases however, as for example where a well defined vertical contact separates a poor conductor from a very good conductor, edge effects may be seen in which the readings vary rapidly with position and are no longer a good indicator of the terrain conductivity. Edge effects may also occur where a very good conductor (a few ohm-meters or less) has dimensions of the order of the intercoil spacing, and again the indicated readings may not accurately reflect the true terrain conductivity. In any circumstance where the apparent conductivity varies significantly in a distance which is short compared with the intercoil spacing the possible presence of edge effects or local subsurface conductors must be considered.

Finally, particularly during mid-summer afternoons, electrical static (electromagnetic radiation from local or distant thunderstorms) may cause the meter readings to become noisy. This is usually evidenced by sudden flicks of the meter needle, however in very severe cases the meter may simply wander about an average reading. Should this occur it is recommended that measurements cease until the "spherics" are over, usually later in the afternoon. Similarly, noisy readings may also be noted when making measurements near large power lines.

5. DATA INTERPRETATION

5.1 Uniform Halfspace

The EM31 has been designed to operate over a range of resistivities from a few ohm-meters to thousands of ohm-meters. For smaller resistivities the instrumental response departs from linearity as shown in Fig. 3, where it will be seen that up to one hundred millimhos per meter the indicated conductivity (σ_a) closely approximates the true conductivity. A departure from linearity is evident for true conductivity greater than one hundred millimhos per meter and beyond one thousand millimhos per meter the instrument response decreases with increasing conductivity. As stated in the introduction, it is generally more informative to observe the spatial variations of terrain conductivity rather than the absolute value of conductivity itself. Fig. 3 shows that as long as the terrain conductivity does not fall below five hundred millimhos per meter (2 ohm-meters) the instrument will be adequately sensitive to small changes in conductivity although it does not read the actual value of conductivity accurately. If necessary, Fig. 3 can be used to correct values of indicated conductivity to actual conductivity.

It should be noted that the graph refers to the worst case viz that of the uniform halfspace. If only a portion of the subsurface ground beneath the instrument is of high conductivity as in the case of horizontal layering, the influence of the high conductivity layers will be proportionately reduced and the indicated conductivity will accurately read the "apparent conductivity" as defined in the following section.

INDICATED CONDUCTIVITY

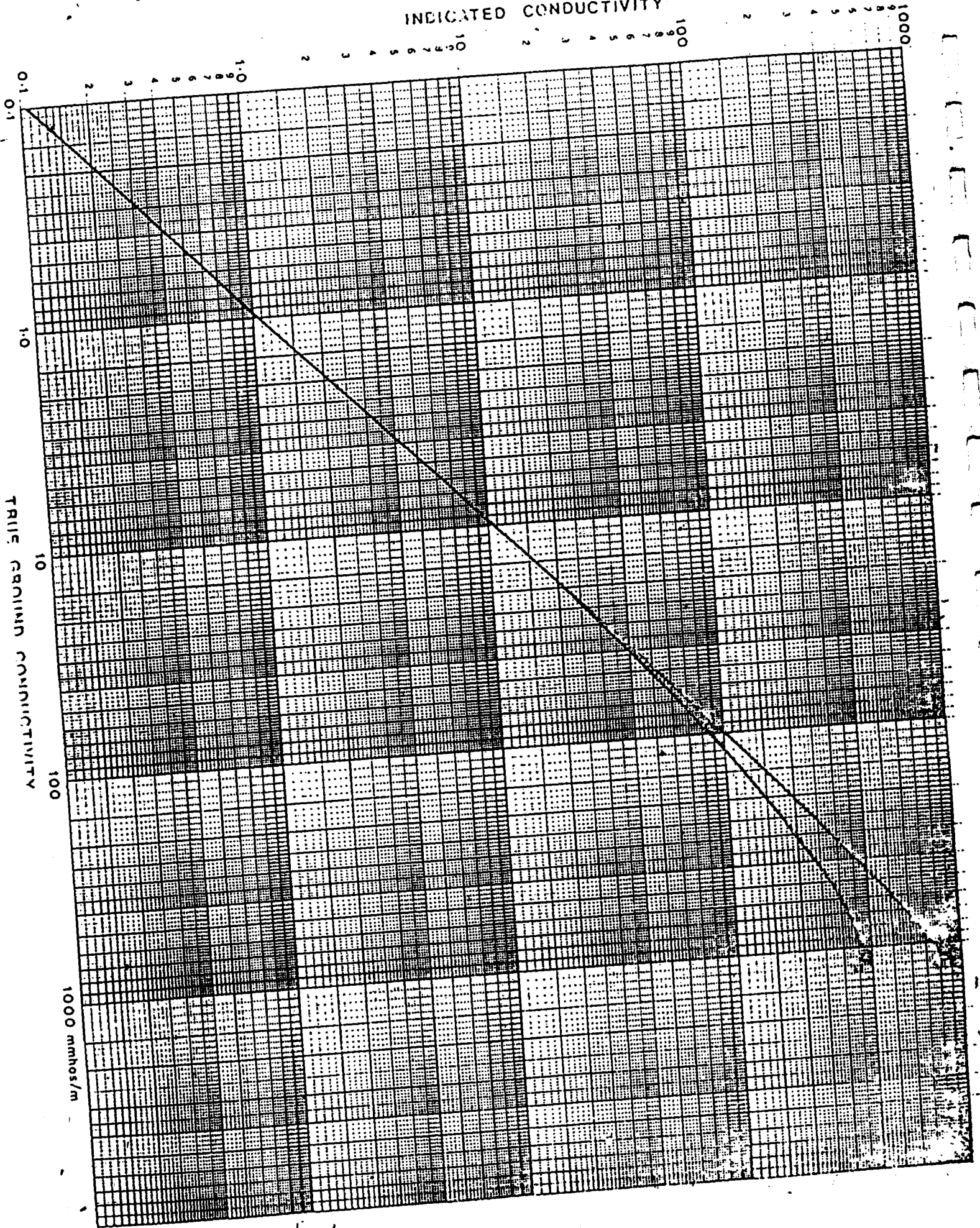


Fig 3

5.2 Multi-Layered Earth

A geophysical model that is of particular importance is the horizontally layered earth, and the EM31 allows a very simple interpretive technique for this model. In order to utilize the model the terrain layering must be well defined and constant over a lateral distance of at least five meters in any direction from the instrument. This condition is often satisfied and this fact is responsible for the usefulness of the technique described herein.

Fig. 4 is a plot of "R(Z)", a function which describes the cumulative relative contribution of all of the material below a depth Z to the instrument reading. Thus if we multiply this function by one hundred (so as to yield percent) all of the ground below a depth of two meters yields 59% of the response, the ground below three meters yields 47%, the ground below six meters yields 29%, etc., assuming that the conductivity itself does not vary with depth.

The algebraic expression given on the figure illustrates the technique which is used to calculate the "apparent conductivity" that will be measured by the instrument for any number of layers, for any values of conductivity and for any thicknesses. Consider for example Fig. 5, which illustrates in cross section a buried river valley cut into shale and subsequently infilled with glacial till. We wish to calculate the instrument response as such a structure is traversed. This is a two layer problem and the expression for the apparent conductivity reduces to:

$$\frac{\sigma_a}{\sigma_1} = 1.000 - R(Z_1) + k_2 R(Z_1) \quad (1)$$

EM 31

MULTI-LAYER RESPONSE DIAGRAM

INST. AT 1 METER FROM GROUND
(NORMAL OPERATING HEIGHT) $R(z)$

1.0

0.9

0.8

0.7

0.6

0.5

0.4

0.3

0.2

0.1

0

1

2

3

4

5

6

7

8

9

$$\frac{R_1}{G_1} = 1.000 = R(z_1)$$

$$+ k_2 [R(z_1) - R(z_2)]$$

$$+ k_3 [R(z_2) - R(z_3)]$$

$$+ \dots + k_n [R(z_{n-1}) - R(z_n)]$$

where

$$k_2 = \frac{G_2}{G_1}, \quad k_3 = \frac{G_3}{G_1}, \quad \text{etc.}$$

SURFACE

 G_1 z_1 G_2 z_2 G_3 z_3 G_{net} z_n

z (meters)

Fig. 4

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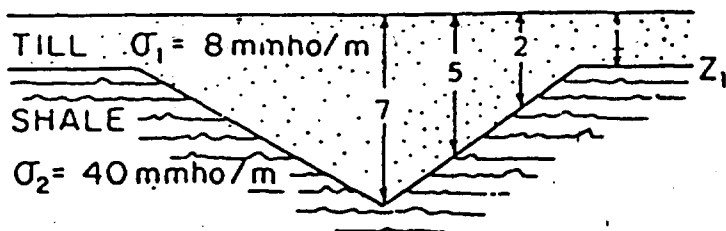
K-2 17 X 10 CM. PER
KEUFFEL & ESSER CO. MADE IN U.S.A.

5.2 Multi-Layered Earth (cont'd)

The table accompanying the figure shows the calculations which yield the value of apparent conductivity at various thicknesses of the upper layer. Thus as we traverse such a structure we would find that the apparent conductivity fell from 32.6 millimhos per meter at a large distance from the buried river valley to 16.0 millimhos per meter at the center of the valley.

Fig. 6 illustrates a second situation where now the objective is to locate bedrock highs within muskeg. This is again a two layer case and the table illustrates the calculations to determine the apparent conductivity. For this configuration, i.e. a conductor on top of an insulator and k_2 a small quantity, the measured apparent conductivity becomes relatively independent of the actual value of the conductivity of the lower layer. For $k_2 \ll 1$, the apparent conductivity simply becomes a function of the thickness of the upper conductive layer.

Finally a third example is given in Fig. 7. In this case we wish to traverse a thickening gravel deposit; the maximum thickness is such that the instrument still responds to material below the deposit. This is an example of the situation of an insulator sandwiched between two conductors and is inevitably the most difficult geometry for electromagnetic systems to detect, as evidenced by the tabulated values of apparent conductivity. Even at a thickness of five meters (station D) there is still significant response from the till beneath the deposit and this response tends to keep the apparent conductivities high.

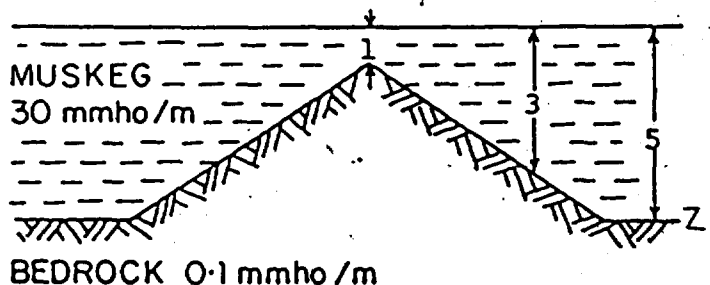


$$\frac{\sigma_a}{\sigma_1} = 1.000 - R(Z_1) + k_2 R(Z_1) \quad (1)$$

$$k_2 = \frac{\sigma_2}{\sigma_1} = \frac{40}{8} = 5$$

$$\begin{aligned} Z_1 = 1\text{m}, \sigma_a &= 8(1.00 - 0.77 + (5 \times 0.77)) = 32.6 \text{ mmho/m} \\ Z_1 = 2\text{m}, \sigma_a &= 8(1.00 - 0.59 + (5 \times 0.59)) = 26.9 \text{ "} \\ Z_1 = 5\text{m}, \sigma_a &= 8(1.00 - 0.33 + (5 \times 0.33)) = 18.6 \text{ "} \\ Z_1 = 7\text{m}, \sigma_a &= 8(1.00 - 0.25 + (5 \times 0.25)) = 16.0 \text{ "} \end{aligned}$$

Fig. 5 CROSS SECTION BURIED RIVER VALLEY

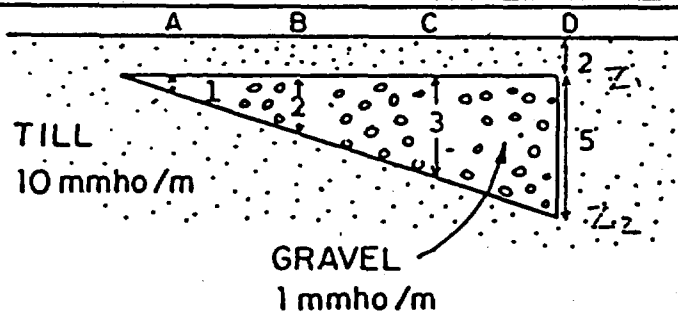


$$\frac{\sigma_a}{\sigma_1} = 1.000 - R(Z_1) + k_2 R(Z_1)$$

$$k_2 = \frac{0.1}{30} = 0.0033$$

$$\begin{aligned} Z_1 = 5\text{m}, \sigma_a &= 30(1.00 - 0.33) = 20.1 \text{ mmho/m} \\ Z_1 = 3\text{m}, \sigma_a &= 30(1.00 - 0.47) = 15.9 \text{ "} \\ Z_1 = 1\text{m}, \sigma_a &= 30(1.00 - 0.77) = 6.9 \text{ "} \end{aligned}$$

Fig. 6 CROSS SECTION BEDROCK HIGH



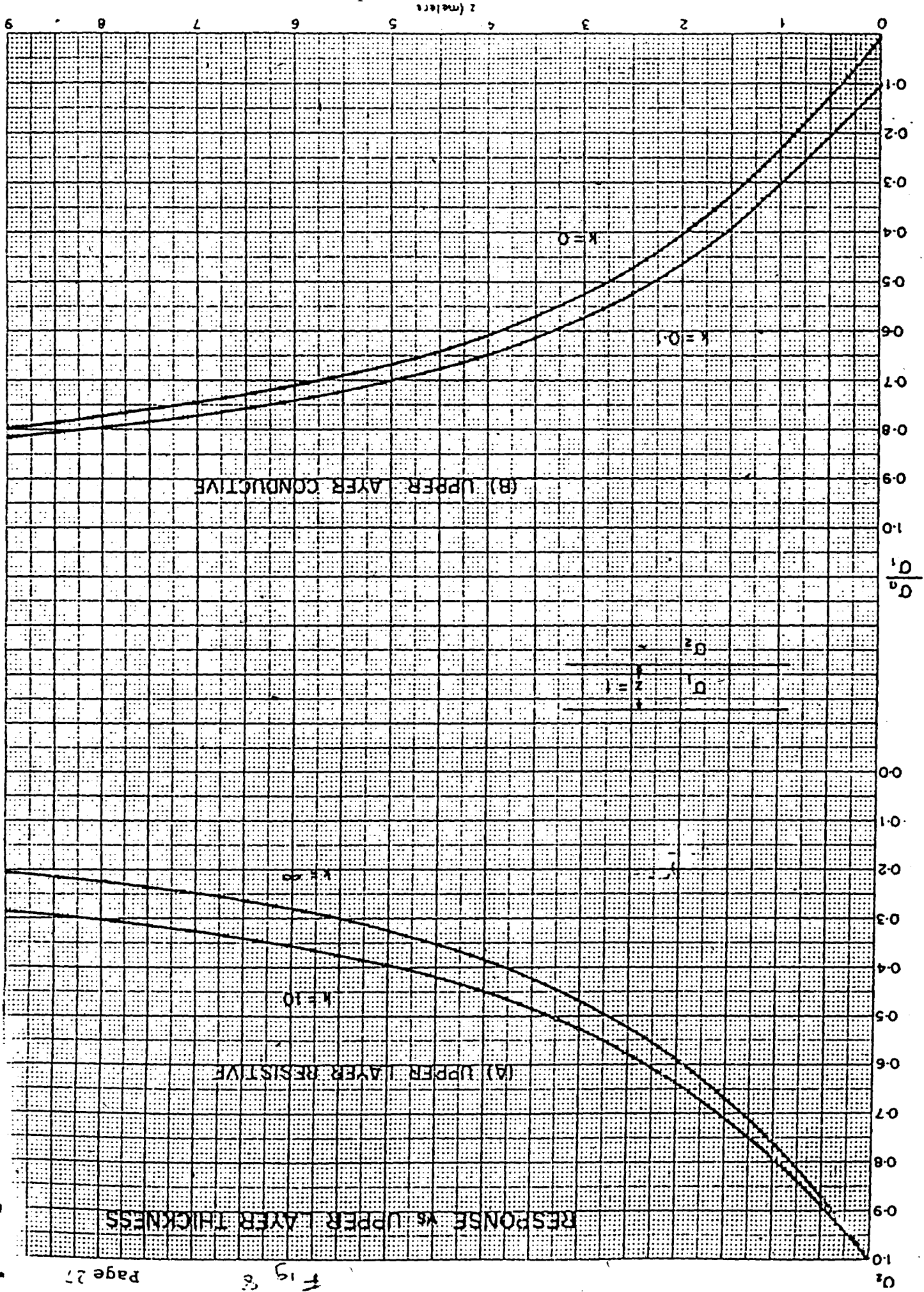
$$\sigma_a = \sigma_1 [1.000 - R(Z_1) + k_2 R(Z_1) - k_2 R(Z_2) + k_3 R(Z_2)]$$

$$k_2 = \frac{\sigma_2}{\sigma_1} = \frac{1}{10} = 0.10$$

$$k_3 = \frac{\sigma_3}{\sigma_1} = 1.00$$

$$\begin{aligned} (A) \sigma_a &= 10 (1.00 - 0.59 + (0.10 \times 0.59) - (0.10 \times 0.47) + (1.00 \times 0.47)) = 8.9 \text{ mmho/m} \\ (B) \sigma_a &= 10 (1.00 - 0.59 + (0.10 \times 0.59) - (0.10 \times 0.39) + (1.00 \times 0.39)) = 8.2 \text{ "} \\ (C) \sigma_a &= 10 (1.00 - 0.59 + (0.10 \times 0.59) - (0.10 \times 0.33) + (1.00 \times 0.33)) = 7.7 \text{ "} \\ (D) \sigma_a &= 10 (1.00 - 0.59 + (0.10 \times 0.59) - (0.10 \times 0.25) + (1.00 \times 0.25)) = 6.9 \text{ "} \end{aligned}$$

Fig. 7 CROSS SECTION GRAVEL DEPOSIT



5.2 Multi-Layered Earth (cont'd)

The three examples show how to calculate the response of the EM31 over a variety of geological environments. Since the instrument operates at a fixed intercoil spacing it is not possible to completely "sound" or evaluate differing conductivity layers with depth. Section 5.3 will show how to further resolve the two-layer case, however it is always useful to be able to calculate the apparent conductivity which would arise from a given multi-layer model to see whether that model fits the measured data.

Fig. 8 shows the apparent conductivity over the two limiting cases of a two layer geometry where the conductivity contrast is very large. In Fig. 8A the upper layer is assumed to be very resistive and the figure shows the apparent conductivity with respect to the lower layer conductivity (assumed known) as a function of the depth below the surface of the lower layer, for values of k of ten and infinity. Thus for large k the figure permits the operator to quickly convert the measured values of apparent conductivity to depth, and to estimate the error if the conductivity contrast is not infinite.

Fig. 8B performs the same task for small values of conductivity contrast. It should be noted that Fig. 8B plots the apparent conductivity with respect to the upper layer conductivity.

It is stated in the data sheet that the effective depth of penetration of the EM31 is approximately six meters. Justification for this claim is shown in Fig. 8 where it is seen that for either a resistive ($k = \infty$) or conductive ($k = 0$) upper layer the apparent conductivity is still

5.2 Multi-Layered Earth (cont'd)

satisfactorily sensitive to changes in the upper layer thickness at six meters. The limitation in resolving further changes in thickness is imposed by probable variations in the conductivity of either layer.

5.3 Geometrical Sounding of Two Layered Earth

It is possible with the EM31 to determine whether the conductivity increases or decreases with depth by laying the instrument on the ground, noting the reading, and then placing the instrument on its side so that the meter face is vertical and again noting the instrument reading. The reason for this is that when the instrument is on its side the new transmitter and receiver coil orientation with respect to the earth causes the effective depth of penetration of the instrument to be essentially halved. It should be noted that in this configuration the current flow does not exhibit the circular symmetry shown in Fig. 1. Thus let σ_a be the apparent conductivity indicated by the instrument on the ground in its normal operating position and let σ_a' be the apparent conductivity indicated when the instrument is lying on its side. If σ_a is greater than σ_a' we know that the conductivity is increasing with depth and vice versa. The ability of the EM31 to indicate whether the conductivity is increasing or decreasing with depth is often of considerable diagnostic value.

It will be observed from the above that it is now possible to obtain two measured quantities at each measurement station. In the event that the earth consists only of two layers, there are three unknown quantities i.e.

5.3 Geometrical Sounding of Two Layered Earth (cont'd)

the conductivity of the upper and lower layer and the thickness of the upper layer. If one of these is known it is possible, using the technique described above, to determine the other two. More importantly, when there is a large conductivity contrast so that the conductivity of either the upper or lower layer can be ignored with respect to the other layer then it is possible to completely resolve the two layered earth. An example of the importance of this can be seen in Fig. 8 which gives the instrumental response as a function of upper layer thickness for either a resistive or conductive upper layer. In order to use these graphs it is necessary to know the conductivity of the lower or upper layer respectively, since both graphs are normalized with respect to these quantities. An inaccurate knowledge of σ_2 in the case of a resistive upper layer or σ_1 in the case of a conductive upper layer can result in a substantial error in the calculated thickness of the upper layer. Any technique which allows us to determine the conductivity of the more conductive layer is of considerable importance since by definition the EM31 is most useful when one layer presents a significant conductivity contrast with respect to the other.

Fig. 9 shows a multi-layer response diagram $R(Z)$, completely analogous to Fig. 4 but with the instrument now laying on the ground. Fig. 10, which presents the function $R'(Z)$ as a function of depth is the same function as Fig. 9 but now with the instrument rotated on its side so that the meter face is vertical. It will be noted from Fig. 9 that with the instrument on the ground in its normal operating configuration the total contribution from

5.3 Geometrical Sounding of Two Layered Earth (cont'd)

all ground in excess of a depth of five meters is 34% whereas Fig. 10 shows that with the instrument lying on its side the ground in excess of five meters causes a total contribution of only 17.5% of the total response. This is the justification for stating earlier that the instrument has effectively one half the depth penetration when lying on its side. Fig. 9 also contains a plot of the ratio R'/R and Fig. 10 a plot of the ratio $(1-R')/(1-R)$, both as a function of depth.

Case 1. σ_1 and σ_2 comparable and σ_1 known. It is shown in Appendix 1 that for this case

$$\frac{R'(Z)}{R(Z)} = \frac{\frac{\sigma_a'}{\sigma_1} - 1}{\frac{\sigma_a}{\sigma_1} - 1}, \quad \sigma_2 = \frac{\sigma_a - \sigma_1 + R\sigma_1}{R'} = \frac{\sigma_a' - \sigma_1 + R'\sigma_1}{R'}$$

thus given σ_a' , σ_a , and σ_1 , R'/R is calculated; Fig. 9 then gives the thickness of the upper layer as a function of R'/R ; with this thickness Fig. 9 or 10 is used to determine R or R' and the second equation given above is used to determine σ_2 thus fully resolving the two layer case.

Case 2. σ_1 and σ_2 comparable and σ_2 known. In this case Appendix 1 shows that

$$\frac{1 - R'(Z)}{1 - R(Z)} = \frac{\frac{\sigma_a'}{\sigma_2} - 1}{\frac{\sigma_a}{\sigma_2} - 1}, \quad \sigma_1 = \frac{\sigma_a - \sigma_2 R}{1 - R} = \frac{\sigma_a' - \sigma_2 R'}{1 - R'}$$

5.3 Geometrical Sounding of Two Layered Earth (cont'd)

Knowing σ_a' , σ_a , and σ_2 , $(1-R')/(1-R)$ is calculated, Fig. 10 gives the thickness and given the thickness Fig. 9 or 10 gives R or R' so that the second equation given above is used to calculate σ_1 .

Case 3. Upper layer resistive. σ_1 much less than σ_2 . In this case Appendix 1 shows that

$$\frac{R'(Z)}{R(Z)} = \frac{\sigma_a'}{\sigma_a}, \quad \sigma_2 = \frac{\sigma_a}{R} = \frac{\sigma_a'}{R'}$$

and the same procedure as for Case 1 above is used to determine the thickness of the upper layer and the conductivity of the lower layer.

Case 4. Upper layer conductive. σ_1 much greater than σ_2 . Again Appendix 1 shows that

$$\frac{1 - R'(Z)}{1 - R(Z)} = \frac{\sigma_a'}{\sigma_a}, \quad \sigma_1 = \frac{\sigma_a}{1 - R} = \frac{\sigma_a'}{1 - R'}$$

and the calculations for Case 2 yield the thickness and the conductivity of the upper layer.

To illustrate the above let it be assumed that the upper layer is known to be 10 millimhos per meter and the EM31 indicates 3.8 millimhos per meter when laying on the ground in its normal configuration and 6.5 millimhos per meter when lying on its side. Fig. 3, which also gives the correction factor to use when the instrument is lying on its side, shows that no correction is necessary.

C. W. 3.1
 σ_a
 C. W. 6.5

5.3 Geometrical Sounding of Two-Layered Earth (cont'd)

Since the instrument has been calibrated to be operated at one meter above the ground both of these numbers must be multiplied by .88 since the instrument is being used on the ground. This results in $\sigma_a = 3.3$ millimhos per meter and $\sigma_a' = 5.7$ millimhos per meter. Then

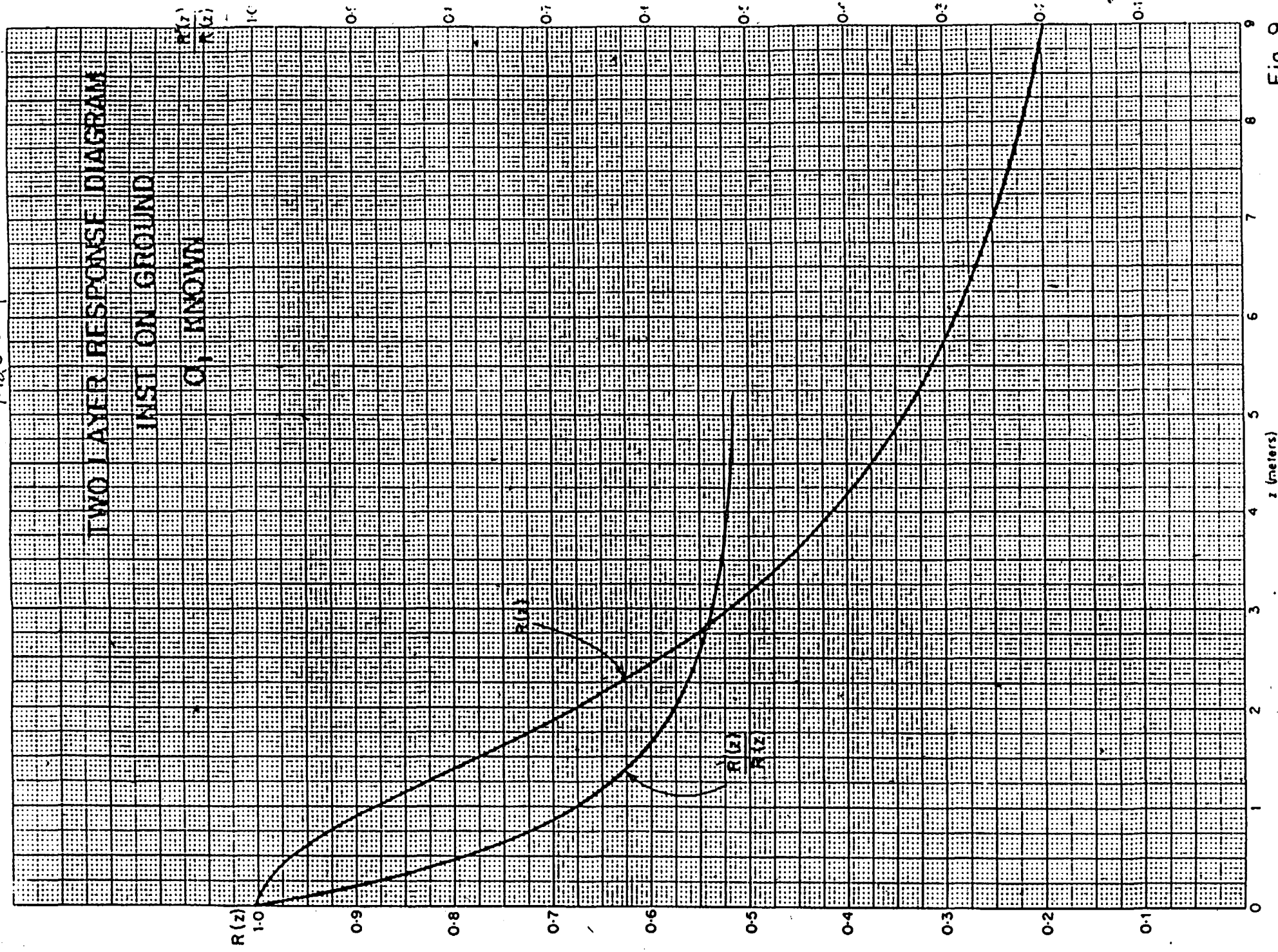
$$\frac{R'(Z)}{R(Z)} = \frac{\frac{\sigma_a'}{\sigma_1} - 1}{\frac{\sigma_a}{\sigma_1} - 1} = \frac{\frac{5.7}{10} - 1}{\frac{3.3}{10} - 1} = 0.64$$

whereupon Fig. 9 shows that the thickness of the upper layer is 1.25 meters and $R(Z) = 0.825$ whereupon

$$\sigma_2 = \frac{\sigma_a - \sigma_1 + R\sigma_1}{R} = \frac{3.3 - 10 + 0.825 \times 10}{0.825} = 1.9$$

The technique for the other cases described above is exactly the same.

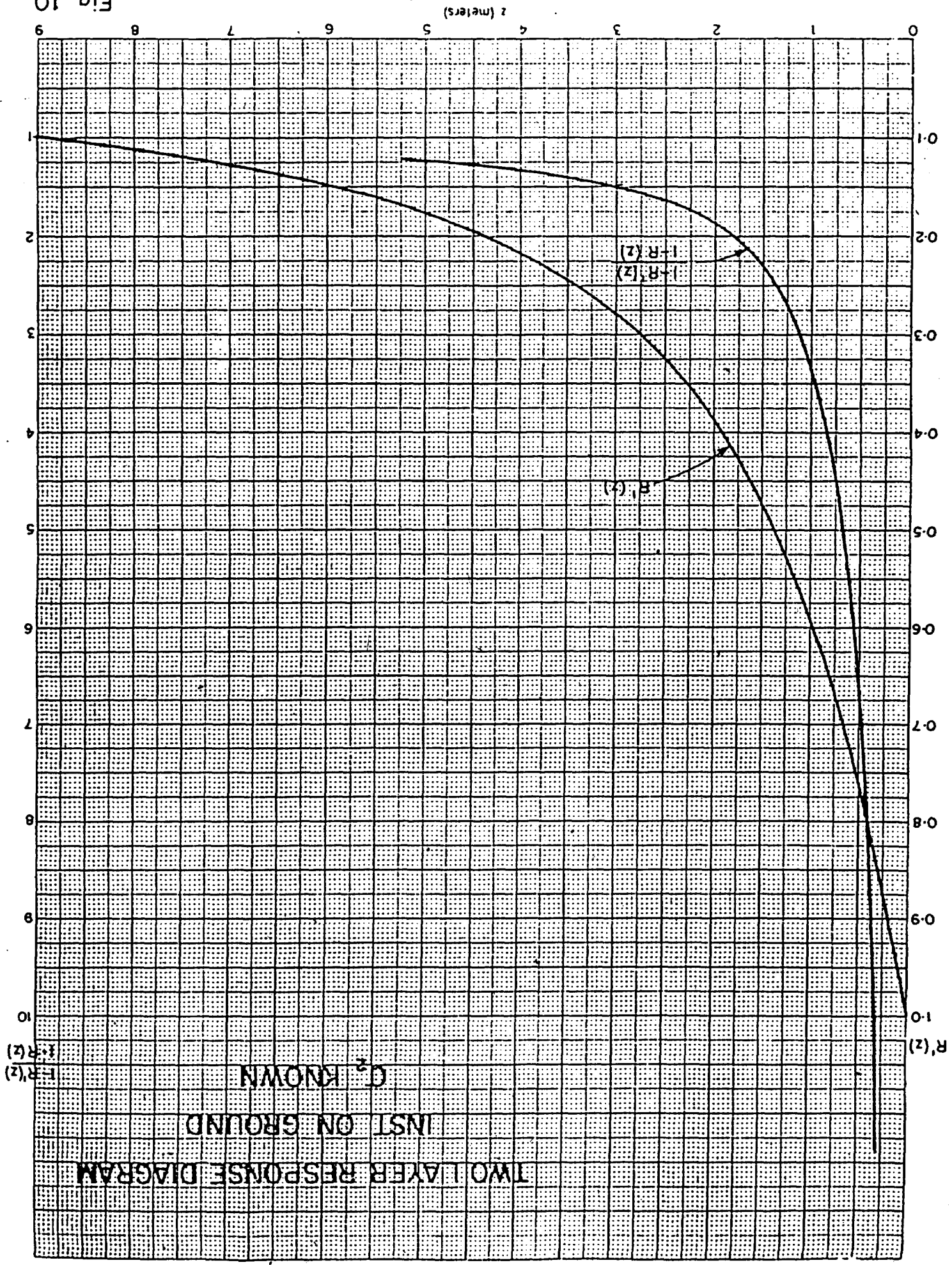
The calculations described above assume that the earth is known to be two-layered. It is possible to determine whether this is true by measuring the apparent conductivity with the EM31 elevated to various heights above the ground and comparing the results with a family of curves given in Appendix II. If the measured variation of apparent conductivity with height matches one of the curves, one can immediately calculate the conductivity of both layers and the thickness of the upper layer, as described therein.



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10 X 10 TO THE CENTIMETER 10 X 25 CM.
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Fig. 10



TWO LAYER RESPONSE DIAGRAM

INST. ON GROUND

Q^2 KNOWN

$1-R'(z)$

$R'(z)$

6. CASE HISTORIES: ELECTROMAGNETIC NON-CONTACTING GROUND
RESISTIVITY MAPPING

6.1 Introduction

This short note gives some illustrative examples of surveys that have been carried out using electromagnetic techniques to measure terrain resistivity.

The instruments employed were the Geonics Limited EM31 and a prototype version of the Geonics Limited EM34. Both instruments were calibrated to read terrain conductivity in millimhos per meter directly; however in some of the case histories illustrated in this note the measurements have been converted to resistivity in ohm-meters.

Two features which often limit the usefulness of conventional ground resistivity surveys are their high cost and in some regions (granular material, frozen ground) difficulties associated with generating sufficient current in the ground. The use of inductive electromagnetic techniques avoids both problems since ground probes are not required. This allows measurement over any type of terrain and greatly reduces the time to perform a survey.

Basically the technique consists of energizing a small coil at an audio frequency and measuring the resultant total magnetic field from this coil and the ground with another coil a fixed distance away. Theoretical studies show that, if the intercoil spacing is maintained at a small fraction of the electrical skin depth in the ground, all of the information about the

6.1 Introduction (cont'd)

ground conductivity is in the quadrature-phase response. Furthermore, the quadrature-phase response is essentially linearly related to ground conductivity, thus permitting an instrument design in which the output is calibrated to read conductivity (or resistivity) directly. Also under these conditions, the effective depth penetration of the system is determined by the intercoil spacing and is independent of skin depth and thus of ground conductivity. This feature greatly simplifies interpretation of survey results. In order to vary the depth to which the resistivity is sensed one simply varies the intercoil spacing in analogy with conventional resistivity surveys. Conversely each survey carried out at a fixed intercoil spacing is essentially analogous to a survey carried out with conventional resistivity equipment at a fixed interprobe spacing.

The Geonics EM31 is a one-man portable instrument designed for engineering geophysical applications down to depths of the order of six meters. The intercoil spacing is fixed at twelve feet (3.66 meters). The effective penetration referred to above is an average value; in those regions where a conductive medium is to be located beneath a resistive layer the penetration depth is substantially larger.

The prototype EM34 is operated at two intercoil spacings viz 50 and 100 feet, resulting in effective depth penetrations of the order of 75 and 150 feet (25 to 50 meters) depending on the intercoil spacing employed for the particular survey. Operation of the EM34 requires two men; however measurements are still taken virtually as fast as the team walks.

6.1 Introduction (cont'd)

Most of the case histories in the technical note have been taken with the EM31; however in some cases data from EM34 surveys are presented in order to further elucidate the features of inductive electromagnetic terrain resistivity mapping.

6.2 Example A: Heart Lake, Ontario (EM31)

Measurement Interval: 100 feet over till,
10 feet over sand/gravel

This survey line compares the results obtained with conventional resistivity equipment (Wenner array with "a" spacings of 1 foot and 20 feet) and the Geonics EM31. It is seen that over the till, where the resistivity is slowly varying, the agreement between the two techniques is excellent. In the region shown as "sand and interbedded gravel" there was a good deal of concretion which caused the resistivity to vary greatly over short distances and which accounts for the discrepancy between the two techniques. Over the till the EM31 was read continuously however the data was recorded, with one exception, at every 100 feet. The exception occurred at station 7+50 where it was noted that a local resistivity high occurred; this was of course not observed on the Wenner array since measurements were taken only at every 100 feet.

6.3 Example B: Sunnybrook Park (EM31, EM34)

Measurement Interval: 100 feet

This case history shows measurements made with both the EM31 and the EM34 and illustrates the effective depth penetration of the two systems. The second sheet shows

6.3 Example B: (cont'd)

the results of expanding Wenner spreads at station 4+00 and 12+00. At station 4+00 we would expect the EM31 to read approximately 40 ohm-meters and the EM34 50 to 60 ohm-meters, which is the case. At station 12+00 the resistivity increases with depth and thus the EM31 should read a relatively low value and the EM34 a higher value which increases with intercoil spacing. This is indeed the case.

6.4 Example C: Cavendish Test Site (EM31)

Measurement Interval: 50 feet

This survey, which was carried out over line C to establish the overburden resistivity shows that with the exception of the swamp area the overburden is extremely resistive. The value of resistivity obtained over the swamp is in good agreement with that from other measurements. The example also illustrates the performance of the instrument over zones A & B, both of which are small highly conductive mineralized zones and cause the instrument to read off scale.

6.5 Example D: Lake Scugog (EM31)

Measurement Interval: 50 feet

This survey illustrates the extremely high resolution achievable with the EM31 or the EM34 systems since neither technique requires electrical contact with the ground. Resolution in conductivity of the order of 3% or 4% is easily achieved and completely repeatable as long as the terrain remains unchanged. The example also illustrates the speed with which a survey can be carried out. In this particular case 1.9 line miles of survey

6.5 Example D: (cont'd)

was performed in seventy minutes with a station interval of 50 feet. Furthermore since the measurements were actually taken continuously any unusual activity in the resistivity between stations would have been recorded.

6.6 Example E: Cooksville/Mississauga, Ontario (EM31)

Measurement Interval: 25 feet

This example illustrates a survey carried out with the EM31 to search for a buried river channel. The channel, which is filled with glacial till, has been cut into the Dundas shale which, as seen from the example, has a resistivity of the order of 25 to 30 ohm-meters. The total time to plot out the profiles shown in the figure was approximately 2½ hours, with a measurement interval of 25 feet. A shortcoming of the technique is seen on line 5, where a region was encountered which was so conductive that it was not possible to take readings.

The second sheet illustrates the application of the two layer curves to interpret the survey results in terms of depth.

6.7 Example F: Discontinuous Permafrost (EM31)

Measurement Interval: Variable

These two examples were taken in Northern Canada and compare the results obtained with the EM31 with a medium frequency version of the Radiohm (Geonics EM16R) technique operating at 250 kHz. The data interval is fairly coarse, nevertheless there is good agreement between the two techniques, which is particularly interesting in view of the fact that the current distribution in the ground

6.7 Example F: (cont'd)

is totally different for the two systems. Furthermore the EM31 operates at a frequency of approximately 40 kHz whereas the MF Radiohm operates at 250 kHz.

6.8 Example G: Pre-glacial River Valley (EM31, EM34)

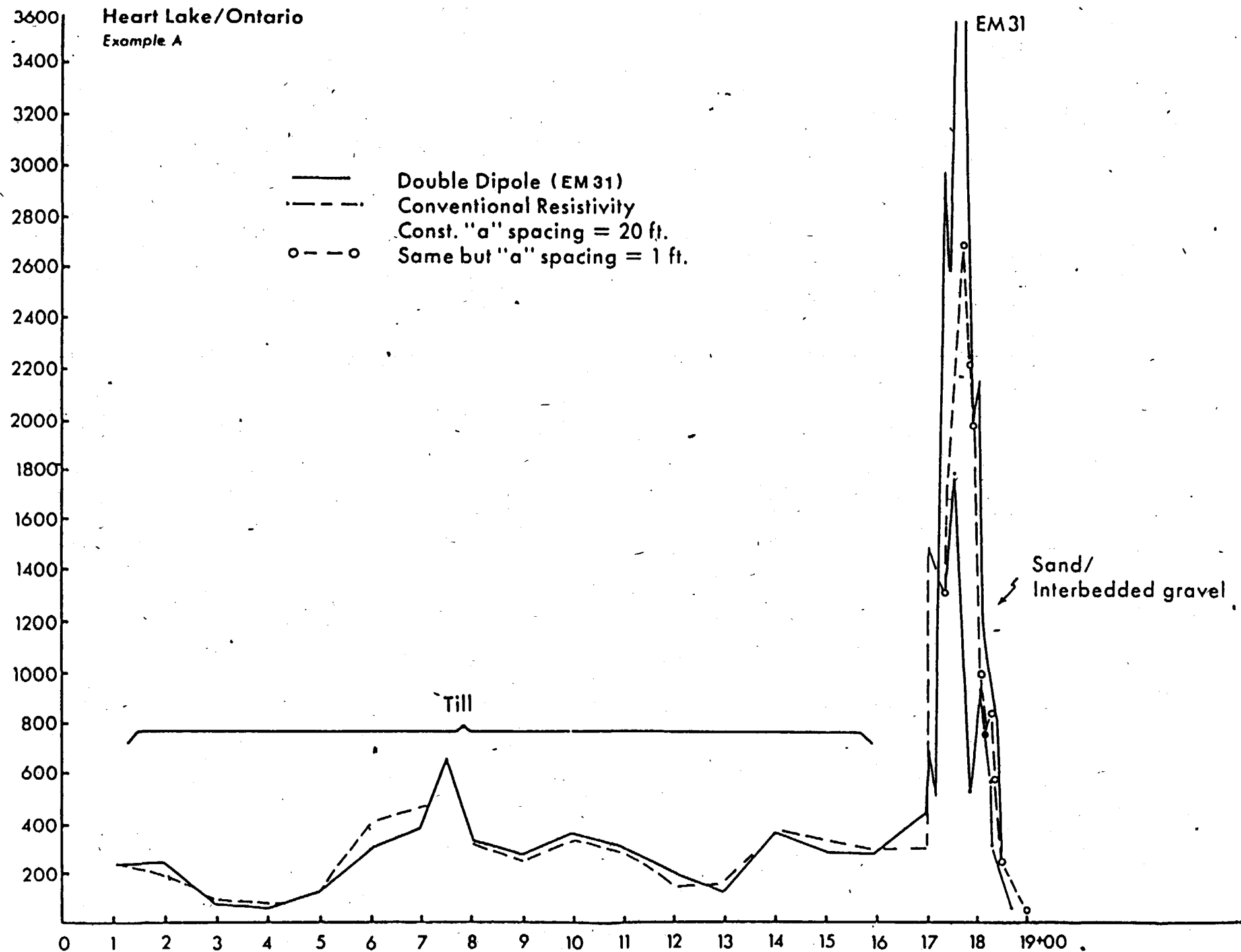
Measurement Interval: 100 feet

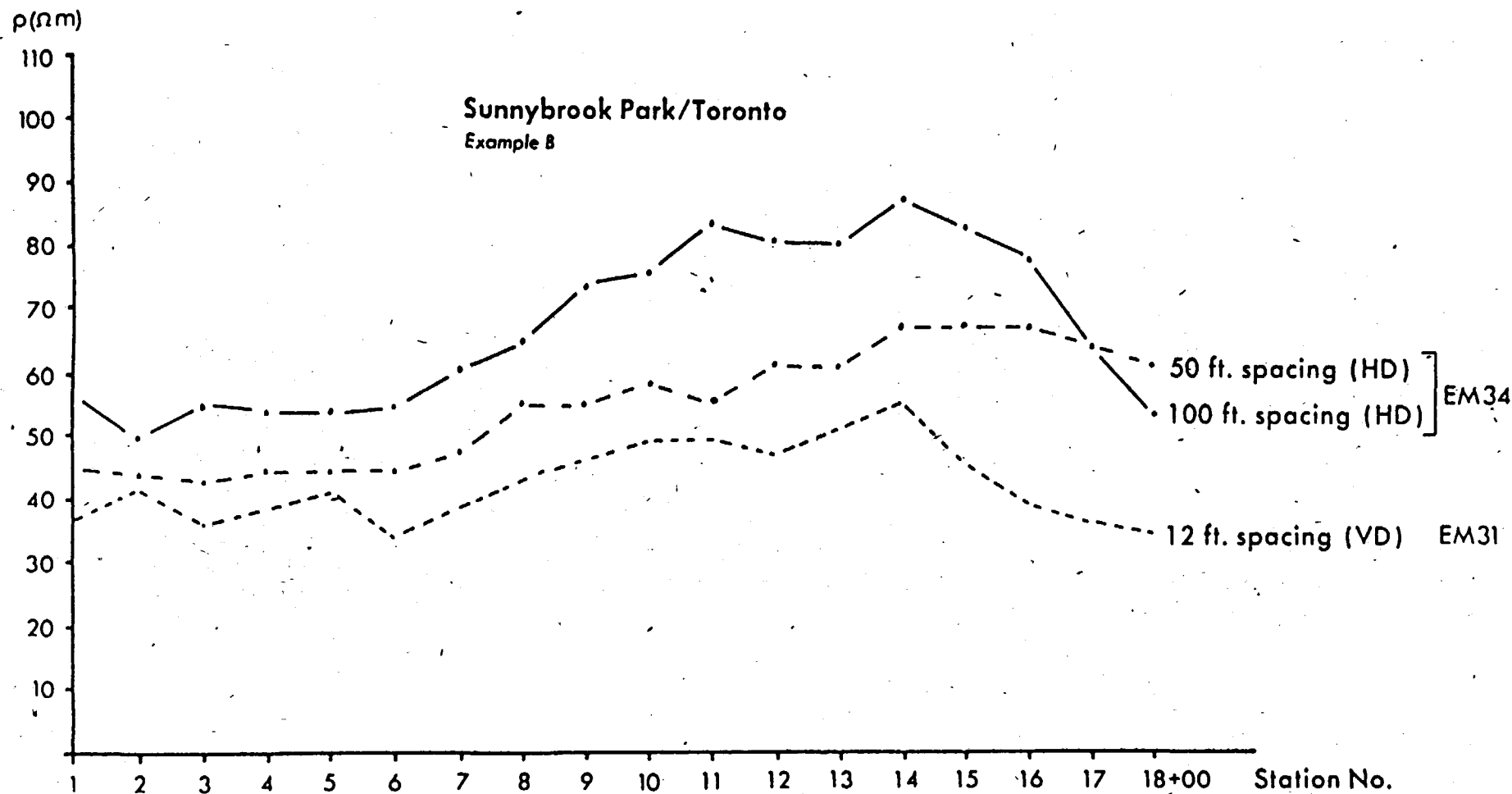
This survey was performed to outline a pre-glacial river valley whose existence had been suggested from water-well data. The survey was performed with the EM34 at a station interval of 100 feet and with intercoil spacing of both 50 and 100 feet. At either spacing the time required to complete the 8400 foot survey line was 1½ hours. The same line was subsequently surveyed with the EM31.

Typical bedrock conductivity in the area is approximately 32 millimhos per meter, whereas an average value for the conductivity of the infilling glacial till is of the order of 8 to 12 millimhos per meter. Thus the EM34 at either intercoil spacing yields approximately 30 to 34 millimhos per meter at the valley edges where the overburden is thin and 12 to 14 millimhos per meter at the valley center. The EM31 yields values of 14 to 18 millimhos per meter at the valley edges (slightly affected by the presence of bedrock) and approximately 10 millimhos per meter at the valley center. The interpreted depth of the valley, based on the model shown in the figure, is approximately 120 feet which is in reasonable agreement with the water-well data value of 150 feet, bearing in mind that the three sets of data show that a two layer model is an over simplification.

The conductivity high which occurs between station 32 and 38 results from a very large pile of waste furnace ash lying on the surface.

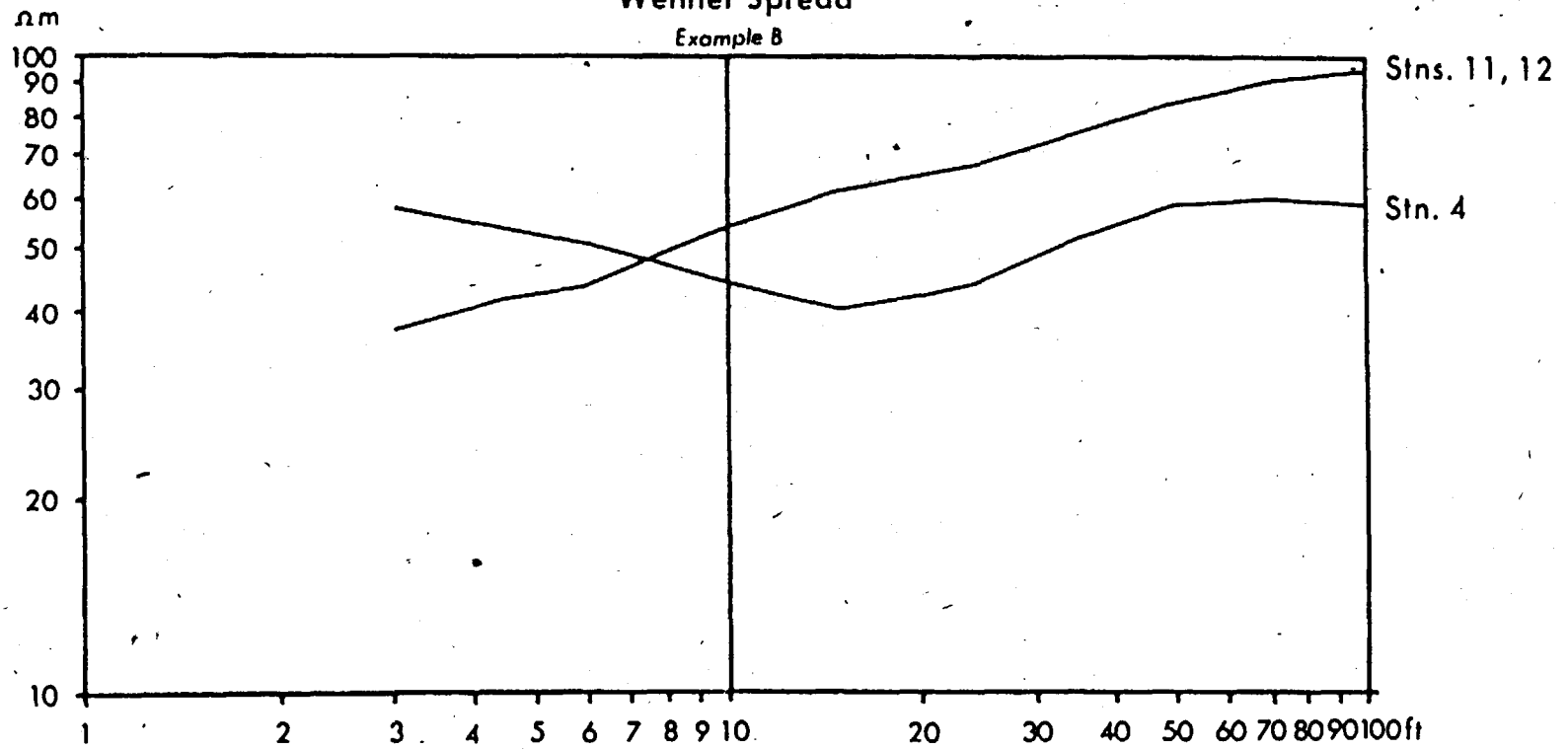
Heart Lake/Ontario
Example A

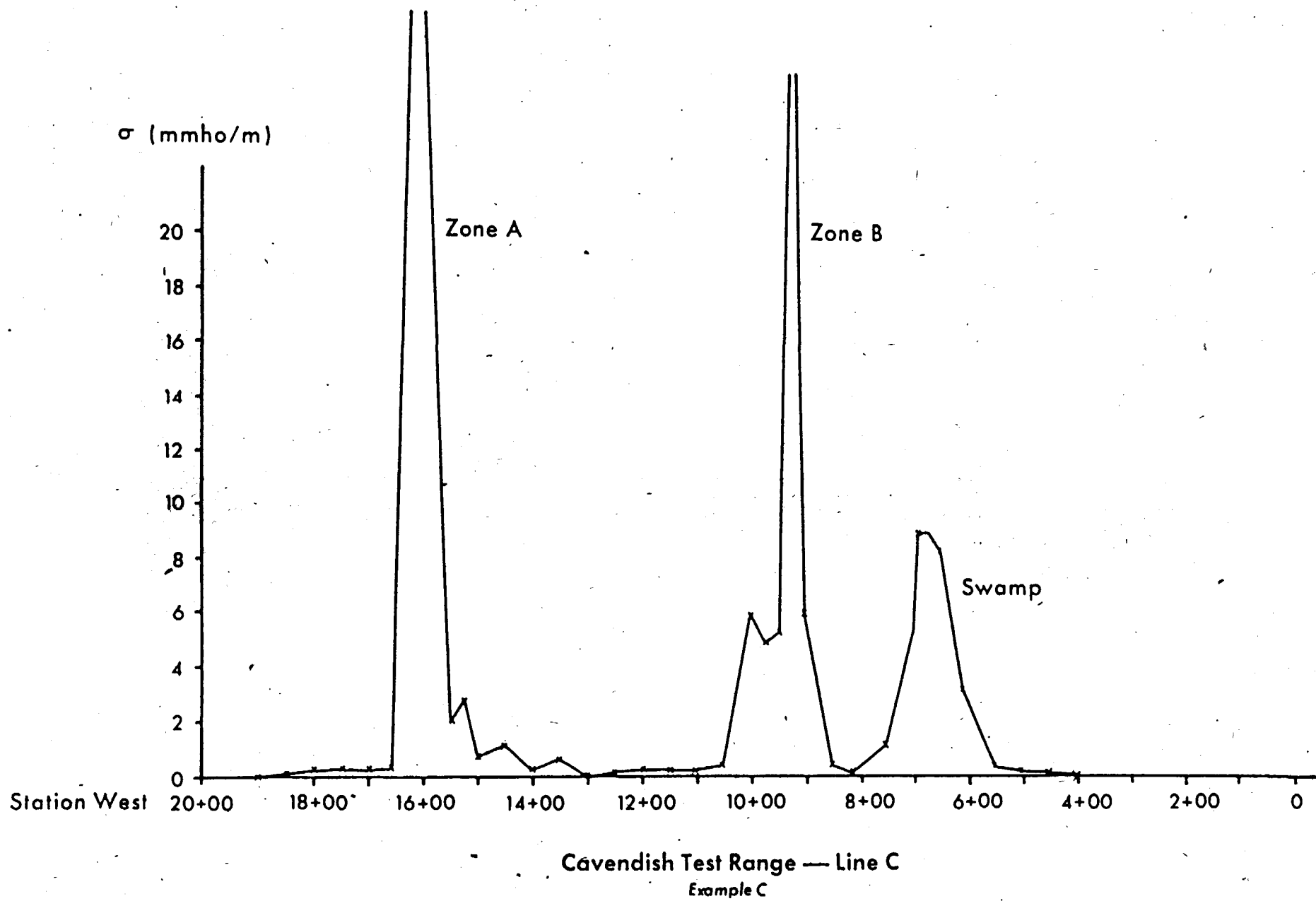




Sunnybrook Park/Toronto Wenner Spread

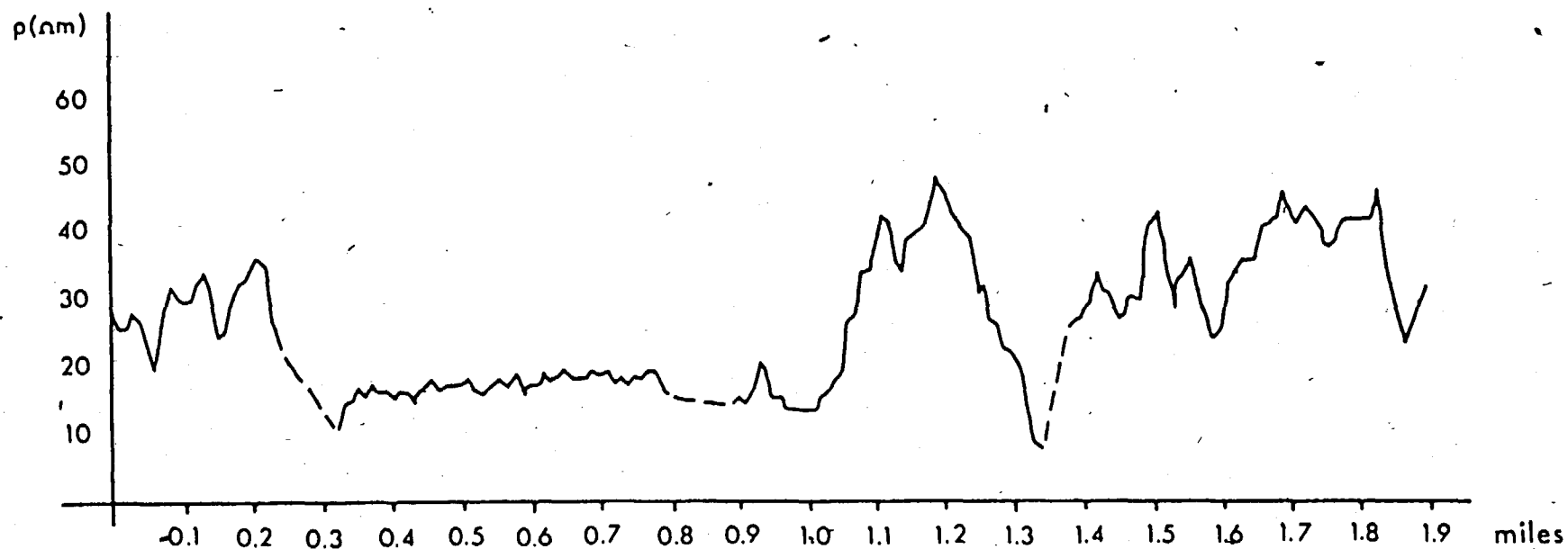
Example B

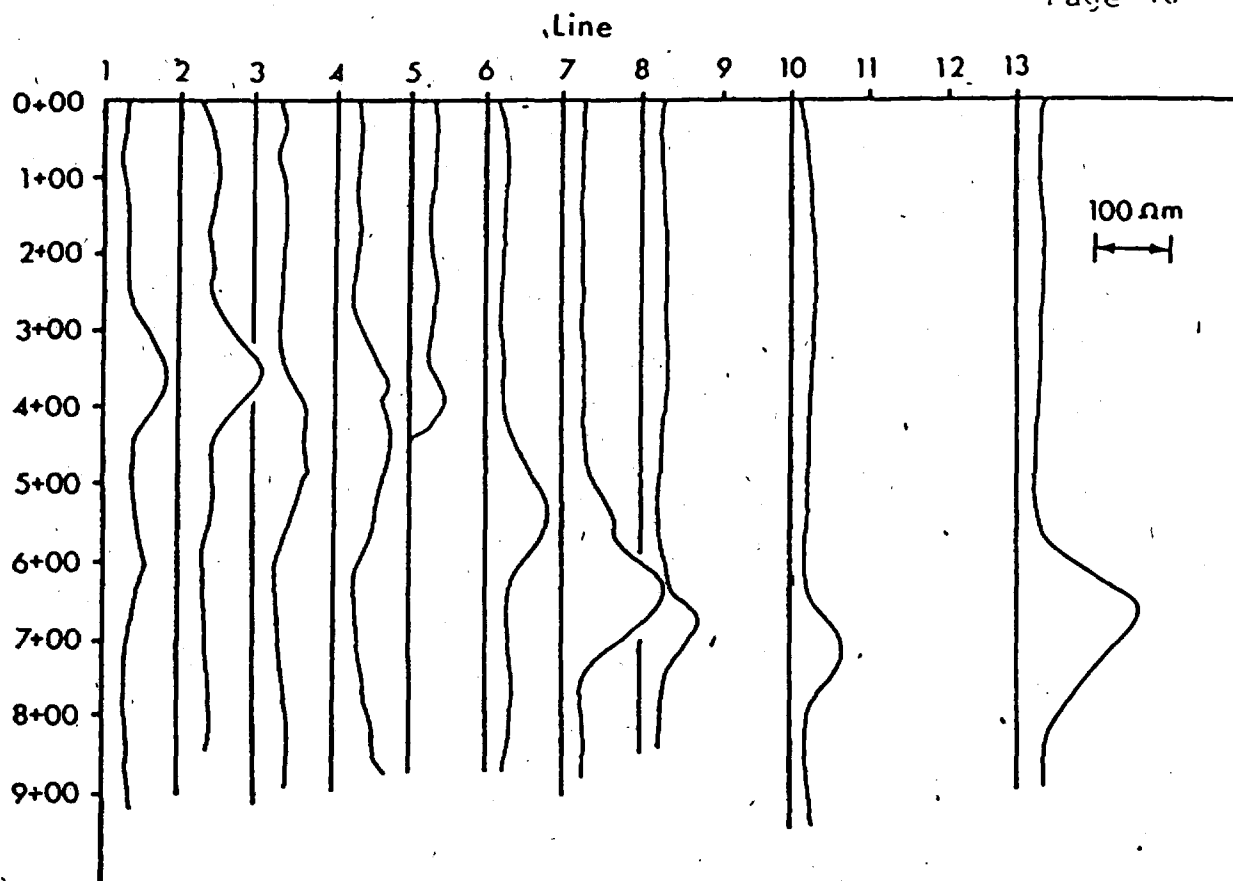




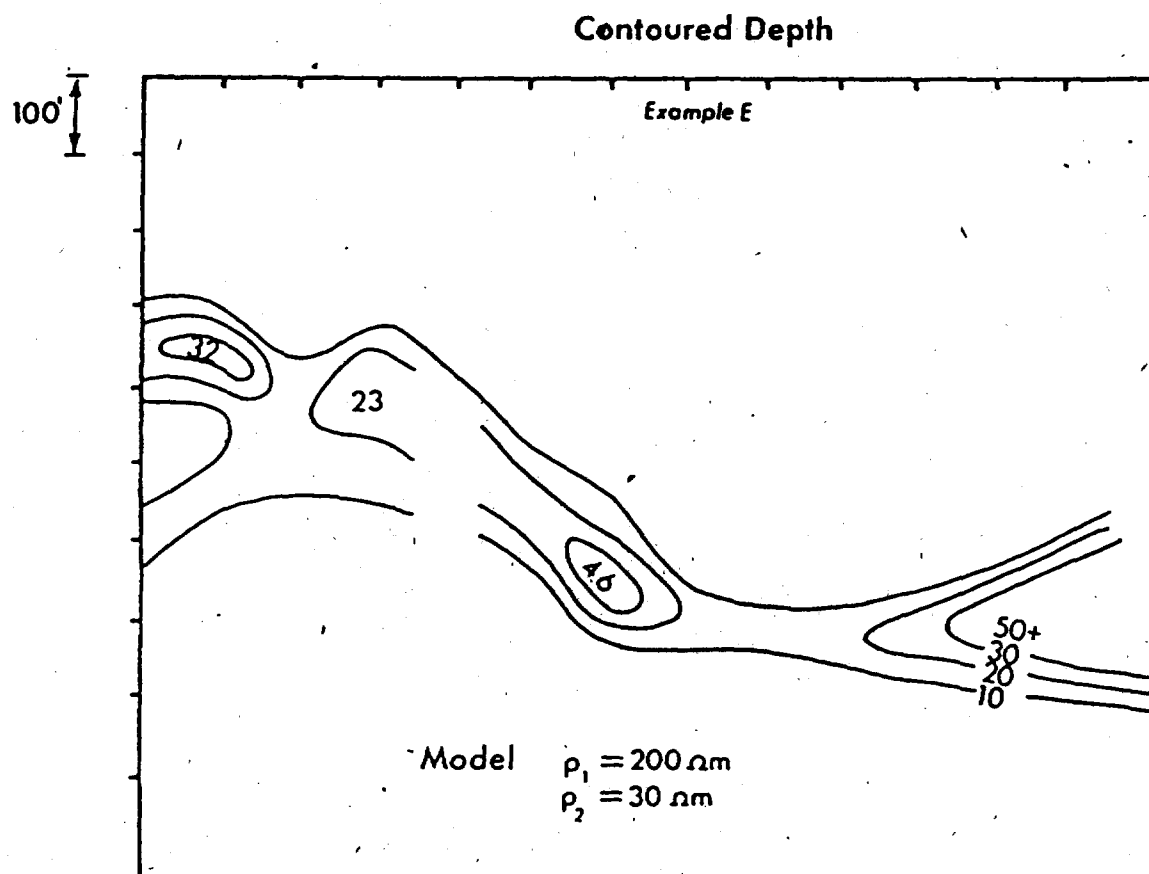
Lake Scugog/East Causeway *Example D*

- Note (1) Station interval = 0.01 mile
(2) Survey duration = 70 minutes
(3) Total no. of stations = 190
(4) 2.7 stations per minute

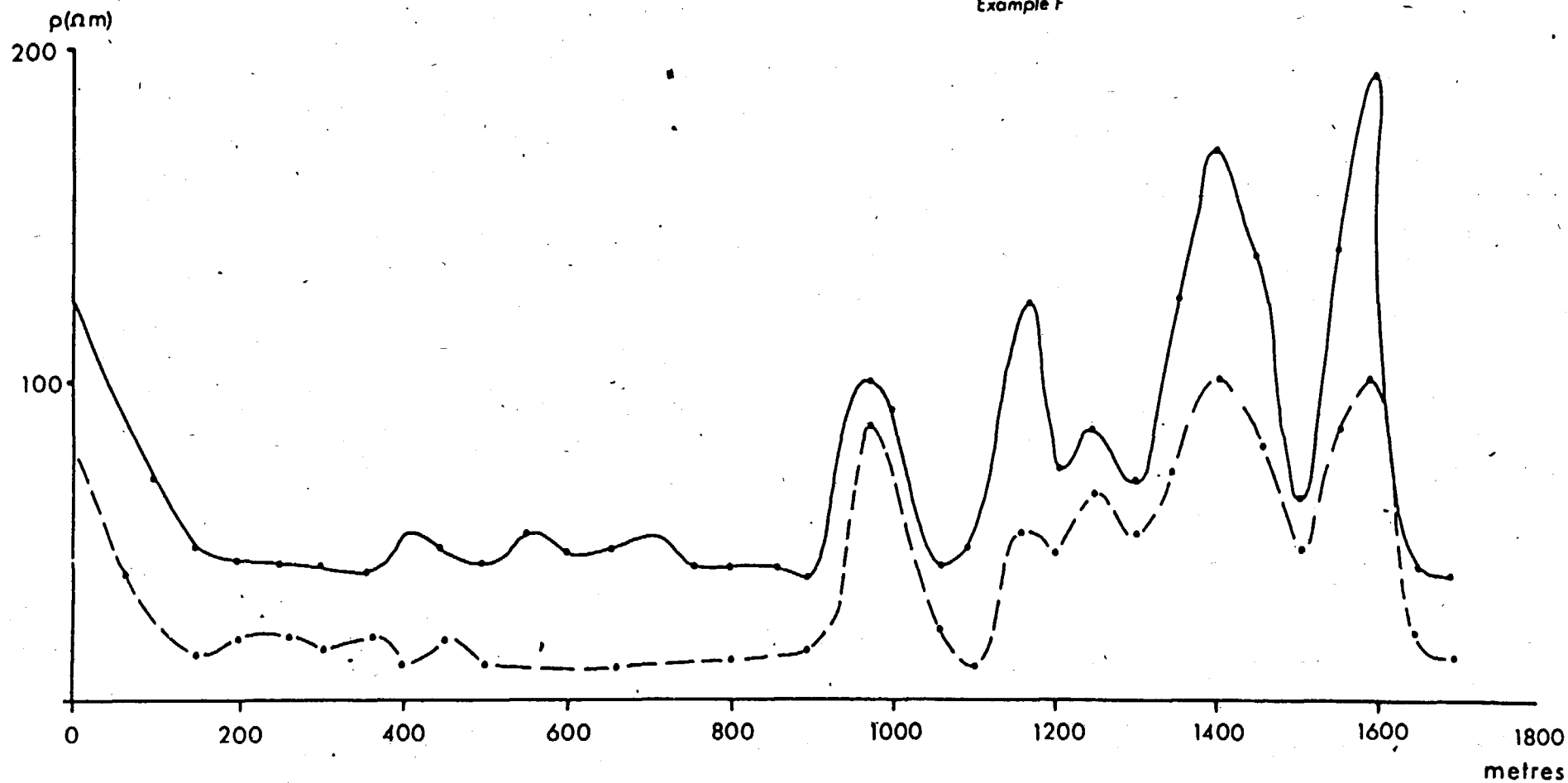




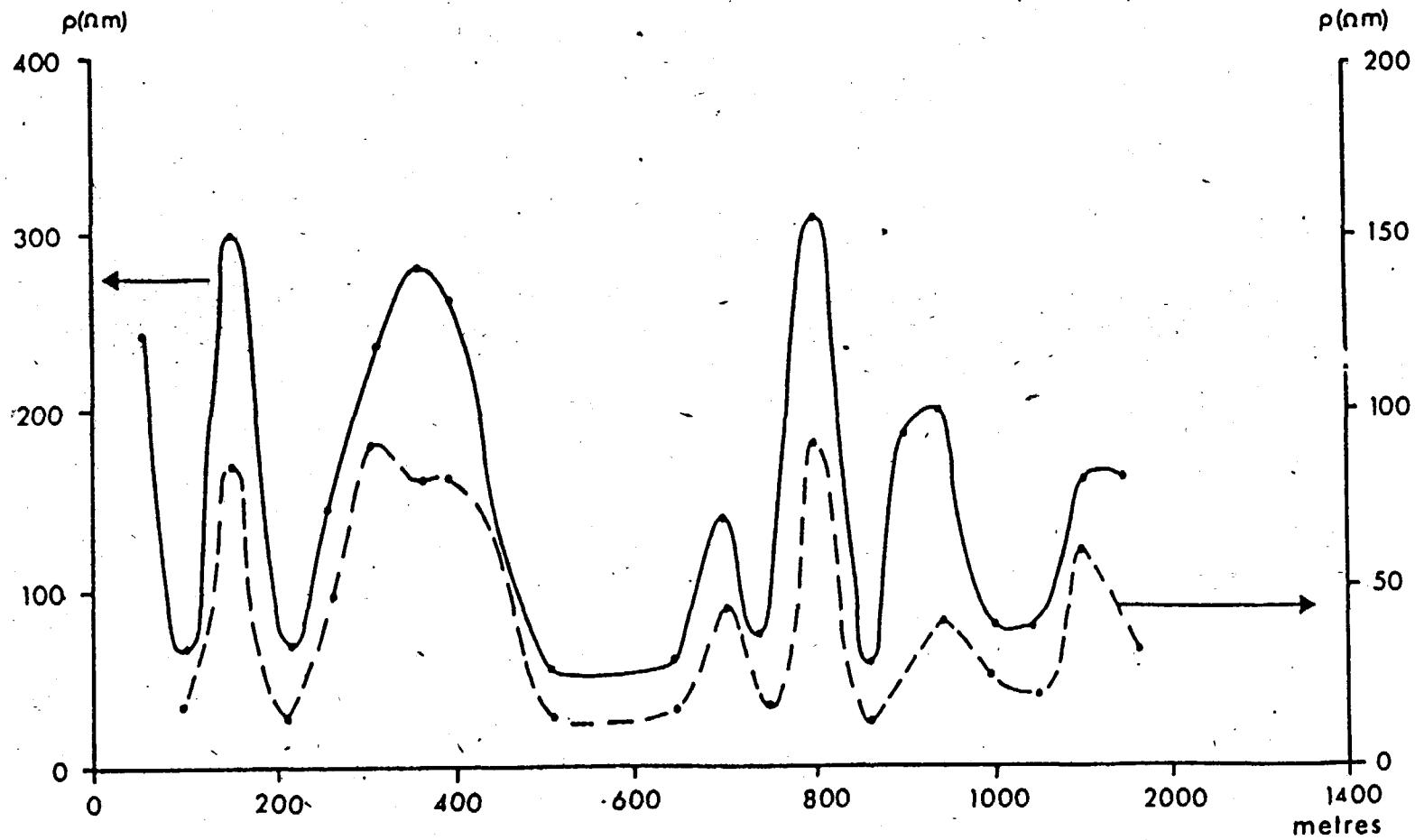
Cooksville/Ontario
Example E

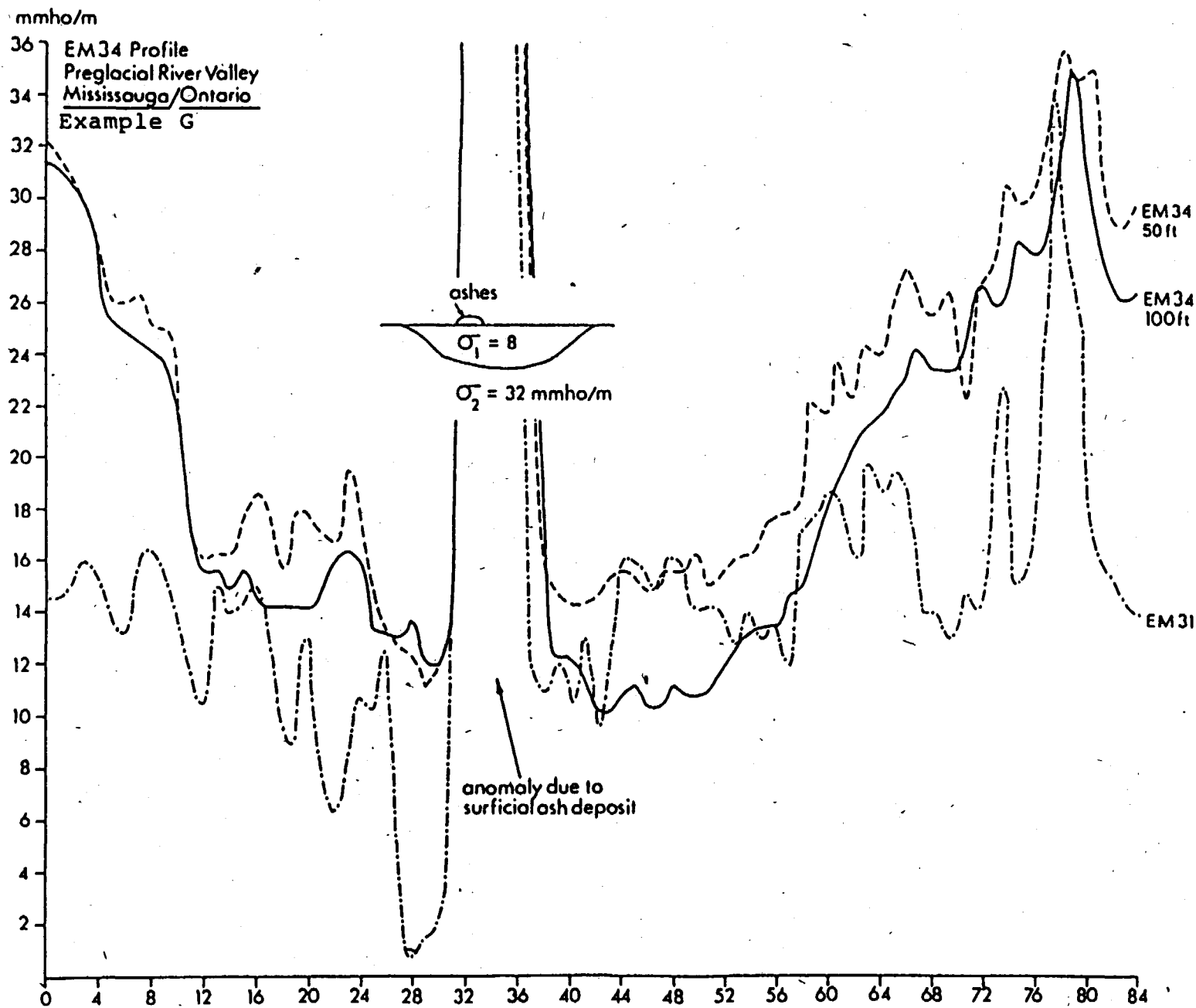


—•— Inductive coupling
- - • - - Plane Wave (LF Radiohm)
Example F



— Inductive coupling
- - Plane Wave (LF Radiohm)
Example F





APPENDIX I - Formulae for Geometrical Sounding of Two-Layered Earth

In the case of a two-layered earth the apparent conductivity is given by:

$$\frac{\sigma_a}{\sigma_1} = 1 - R + kR = R(k - 1) + 1 \quad (1)$$

with the instrument on the ground in its normal operating position, and

$$\frac{\sigma_a'}{\sigma_1} = 1 - R' + kR' = R'(k - 1) + 1 \quad (2)$$

with the instrument on the ground on its side.

* Case 1. Therefore

$$\frac{R'}{R} = \frac{\frac{\sigma_a'}{\sigma_1} - 1}{\frac{\sigma_a}{\sigma_1} - 1} \quad (3)$$

and from the first and/or second equations, with

$$k = \sigma_2 / \sigma_1$$

$$\sigma_2 = \frac{\sigma_a - \sigma_1 + R\sigma_1}{R} = \frac{\sigma_a' - \sigma_1 + R'\sigma_1}{R'} \quad (4)$$

* Case 2. When the conductivity of the lower layer is known, from equations (1) and (2)

$$\sigma_a' = \sigma_1 - R'\sigma_1 + R'\sigma_2 \quad (5)$$

APPENDIX I.- Cont'd

Case 2. (cont'd)

$$\sigma_a = \sigma_1 - R\sigma_1 + R\sigma_2 \quad (6)$$

$$\sigma_a' - \sigma_2 = \sigma_1 - R'\sigma_1 + R'\sigma_2 - \sigma_2 \quad (7)$$

$$= (1 - R')(\sigma_1 - \sigma_2) \quad (8)$$

$$\sigma_a - \sigma_2 = (1 - R)(\sigma_1 - \sigma_2) \quad (9)$$

$$\frac{1 - R'}{1 - R} = \frac{\frac{\sigma_a'}{\sigma_2} - 1}{\frac{\sigma_a}{\sigma_2} - 1} \quad (10)$$

$$\sigma_1 = \frac{\sigma_a - \sigma_2 R}{1 - R} = \frac{\sigma_a' - \sigma_2 R'}{1 - R'} \quad (11)$$

*Case 3. When the upper layer is resistive and σ_2 is much greater, then σ_1 , from equations (5) and (6)

$$\sigma_a = \sigma_2 R \quad (12)$$

$$\sigma_a' = \sigma_2 R' \quad (13)$$

$$\frac{\sigma_a'}{\sigma_2} = \frac{R'}{R} \quad (14)$$

and

$$\sigma_2 = \frac{\sigma_a}{R} = \frac{\sigma_a'}{R'} \quad (15)$$

APPENDIX I - Cont'd

*Case 4. When the upper layer is conductive and σ_2 is much less than σ_1 , from equations (5) and (6)

$$\sigma_a = \sigma_1(1 - R) \quad (16)$$

$$\sigma_a' = \sigma_1(1 - R') \quad (17)$$

$$\frac{1 - R'}{1 - R} = \frac{\sigma_a'}{\sigma_a} \quad (18)$$

$$\sigma_1 = \frac{\sigma_a}{1 - R} = \frac{\sigma_a'}{1 - R'} \quad (19)$$

APPENDIX II - Determination of Two-Layered Earth Geometry by Varying Instrument Height

The electrical conductivity of the earth often exhibits horizontal layering and it is possible with the EM31 to determine how closely this layering can be approximated by a two-layered earth geometry.

Suppose measurements made with the instrument on the ground have yielded different values of apparent conductivity when the instrument meter is (a) face-up (i.e. the instrument is in normal position), and (b) vertical (i.e. the instrument is lying on its side). As discussed in Section 5.3 had the value of apparent conductivity been the same in both positions we would know that the earth was homogeneous, but the fact that the values are different for the two positions shows that the electrical conductivity varies with depth. We would now like to know whether the earth is two-layered.

To determine this we make measurements of the apparent conductivity with the instrument at various heights above the ground, as measured by the distance between the ground and the nearest point of the white tube. It is suggested that measurements be made at increments at least as small as one-half meter, for an example at 0, 0.5, 1, and 1.5 meters. Measurements made at smaller increments will yield greater accuracy, as will measurements made up to 2 meters height if possible. Measurements should be made both with the instrument in its normal position (meter face upright, i.e. vertical dipoles,) and with the instrument on its side (meter face vertical, horizontal dipoles) since it will be seen from the curves that the fall-off with height for these two configurations is quite different and that this difference is therefore of considerable diagnostic value in determining the two-layered earth characteristic. It is important to make the measurements of height and apparent conductivity as accurately as possible.

APPENDIX II - Cont'd

The curves in the two figures show the variation of apparent conductivity (divided by upper layer conductivity) for instrument height varying from 0 to 2 meters and for five values of upper layer thickness viz. 0.5, 1.0, 1.5, 2.0, and 3.0 meters. The relationship is shown for both vertical dipoles (meter face up, instrument in normal position) and horizontal dipoles (meter face vertical, instrument on its side) for various values of upper layer to lower layer conductivity contrast.

To use the curves one proceeds as follows:

- (1) Place a piece of semi-transparent paper over either one of the graphs and on it plot the measured values of apparent conductivity versus instrument height to the scale used on the figures. Also sketch in on the transparent paper at any point a horizontal or vertical line to insure that when the transparent paper is translated over the figures in step 2 it does not rotate.
- (2) Translate the transparent paper vertically and horizontally (i.e. without rotation) across the various plots shown on the figures to see whether the data points can be matched to one of the curves. In the event that no satisfactory match is achieved the earth cannot be represented by a two-layered case.
- (3) If a satisfactory match can be achieved, the parameters of the two-layered earth are immediately calculated as follows: suppose that the measured values of apparent conductivity with height are

Height =	<u>0.0</u>	<u>0.5</u>	<u>1.0</u>	<u>1.5</u>	<u>2.0</u> meters
σ_a (EM31 upright)	20.7	20.7	19.3	17.1	14.1 mmho/m
σ'_a (EM31 on side)	26.0	19.0	14.3	11.2	9.0 mmho/m

APPENDIX II - Cont'd

Upon plotting these data values to the scale of the figures and translating them we see that best agreement is achieved for an upper layer thickness of 1.5 meters and a conductivity contrast $k = 0.5$. We further observe that the value of 20.7 millimhos per meter for σ_a (instrument up-right) corresponds to a value of σ_a/σ_1 of 0.62.

$$\text{Since } \frac{\sigma_a}{\sigma_1} = 0.62 \text{ when } \sigma_a = 20.7$$

$$\therefore \sigma_1 = \frac{20.7}{0.62} = \underline{33.4} \text{ mmho/m}$$

$$\text{Since } k = \frac{\sigma_2}{\sigma_1} = 0.5$$

$$\therefore \sigma_2 = 0.5 \times 33.4 = \underline{16.7} \text{ mmho/m}$$

$$\text{and } t = \underline{1.5m} \text{ from the figure.}$$

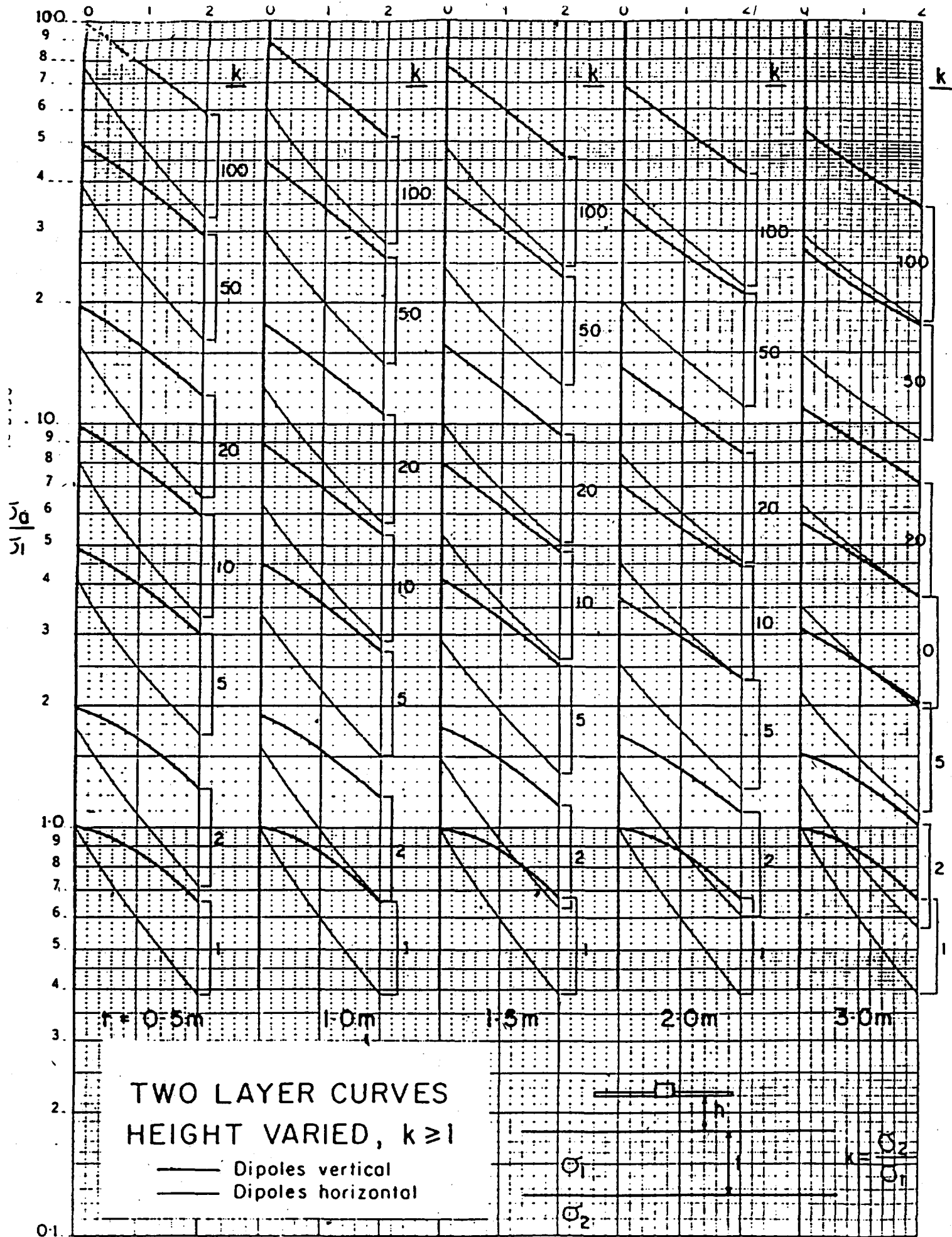
The two-layered earth is fully resolved.

It will be observed that in some regions on these two figures the resolution or differentiation between the various curves is not very great. For example if one examines the curves for $t = 1.0$ meters, and $k = 100, 50$, or 20, one sees that these curve pairs are almost identical. If the process described in step 3 is carried out for each of these conductivity contrasts the result of the calculations will show that the lower layer conductivity and the upper layer thickness are the same, regardless of which of the three curve pairs are used, and that the only calculated parameter that will vary will be the upper layer conductivity which is very small. This is an example of the well-known property of electromagnetic systems to accurately give both the distance to a good conductor and

APPENDIX II - Cont'd

the actual conductivity of the conductor but to poorly define the intervening resistive material. A problem in unambiguously matching the curves may also occur for larger values of upper layer thickness, where the thickness is sufficiently great to prevent accurate resolution of its value.

These features notwithstanding it will be found that these curves are useful in (a) deciding whether the ground resembles a two-layer case and if so (b) giving a reasonably accurate estimate of the electrical parameters.



Appendix A-A2

Dual Tube Reverse Circulation Air Rotary Drilling Methods

DUAL-WALL REVERSE CIRCULATION OFFERS.

HOW IT WORKS

The reverse circulation rotary drilling method utilizes dual-wall pipe, top drive rotation and a side inlet for injecting the drilling fluid, air or both.

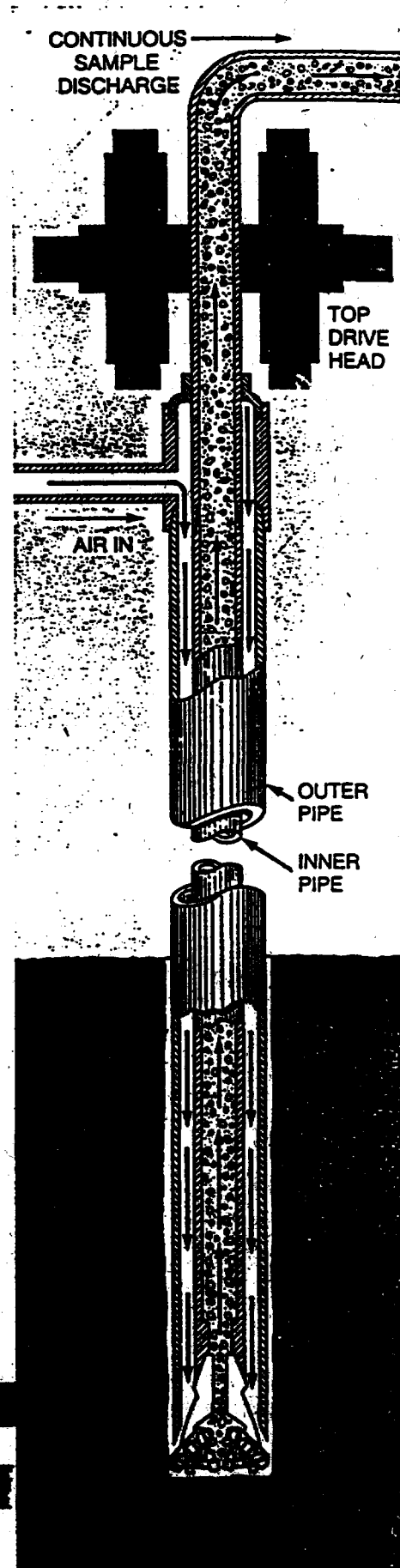
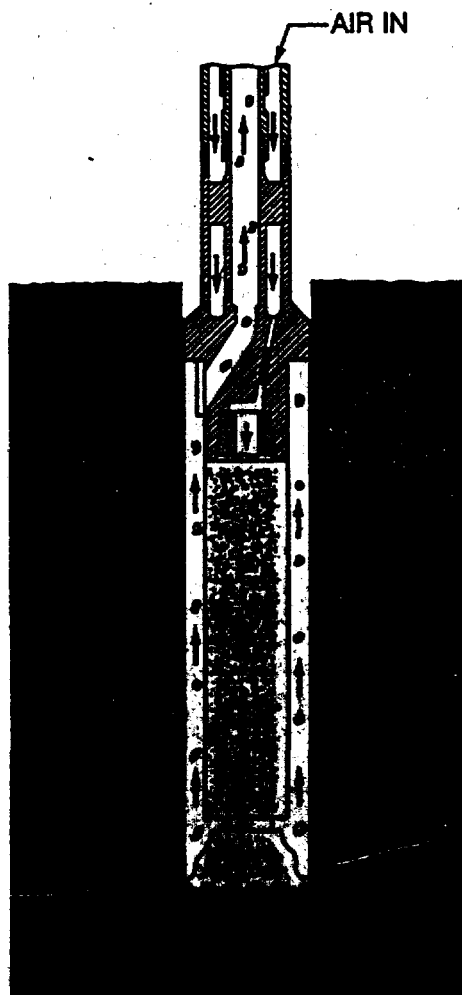
When drilling with this system, the drilling fluid is forced down the outer annulus of the dual-wall pipe to the drill bit where it is then directed to the center of the pipe. The air or drilling fluid returns the cuttings via the inner pipe at velocities in excess of 4,000 feet per minute.

The reverse circulation rotary system uses flush-jointed drill pipe and a drill bit sub designed to fit snugly over the body of the drill bit. This unique design permits the bore hole to be cut with a minimum of clearance and so lessens the possibility of sample contamination. This configuration also minimizes the loss of air or misting in vuggy or fractured formations and will maximize the geological sample recovery in adverse conditions; conditions that are nearly impossible for rotary or diamond drills.

FOR HARD ROCK FORMATIONS

When it becomes uneconomical to drill in hard formations with either a tricone or roller button bit, the dual-wall method can still be used to advantage with a down-the-hole hammer drill.

An interchange sub is screwed onto the top of the hammer drill and then the complete unit is screwed onto the dual-wall pipe. In this configuration, the rotating/percussion action of the drill cuts the sample which travels between the wall of the hole and the outer barrel of the hammer drill. As the sample moves up the length of the hammer drill it is directed into the interchange sub openings where it is air lifted to the surface through the inner pipe.



EFFICIENCY, VERSATILITY AND ECONOMY

HOW LAYNE MAKES IT WORK FOR YOU

Whether you call it the double-wall pipe system, rotary continuous sample system or the dual-wall system, the results are the same . . . Layne can provide you with a faster, less costly way to obtain the purest exploration samples possible.

Uncontaminated Sampling

Since rock and water samples do not come in contact with the bore hole, there is no contamination from the formation or erosion. In extreme high water bearing formations, down hole erosion may occur due to fractures or gravel formations.

Faster Penetration

With top head drive rotary drills, exploration proceeds up to 15 times faster than with conventional core drills.

Lower Operating Costs

In some cases, costs have been reduced by 75%. Bit costs are generally lower and the job is completed in less drilling time. In

addition, expensive drilling muds or additives are rarely used.

Straighter Holes

Flush-wall pipe with minimum clearance results in less deviation than with conventional drilling methods.

No Surface Casing

The outer pipe supports the bore hole while circulation is maintained internally.

No Lost Circulation

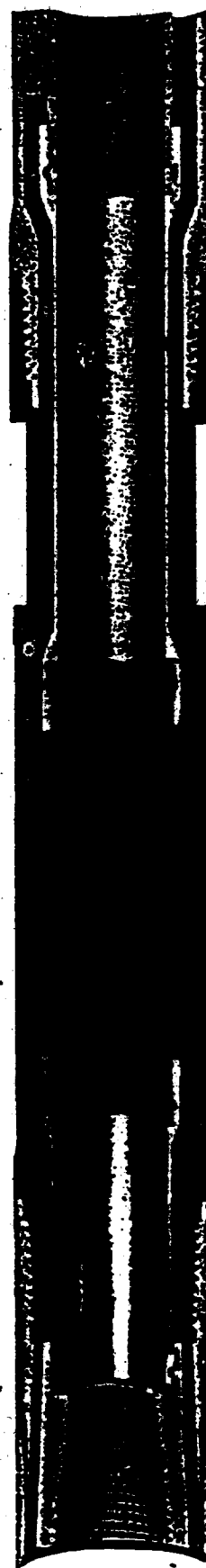
Circulation is maintained even when drilling in vugs, fractures, voids and joints thus avoiding expensive delays.

Improved Well Design

Better samples provide greater reliability in well design, enhancing results.

In-Place Water Sampling

Many geologic environments are suitable for obtaining reliable water samples from aquifer zones during drilling operations, eliminating the need for expensive packers or other methods of obtaining such samples.



TYPICAL SCENARIO FOR DUAL-WALL REVERSE CIRCULATION DRILLING AND BORING PROGRAM

The general purpose for completing the soil boring is to provide detailed characterization of subsurface hydrogeologic conditions within the surficial unconsolidated aquifer materials (soils above bedrock) and, at some locations, characterization of the bedrock surface. Specific program objectives include the following:

1. Obtain continuous soil samples for purposes of characterizing and classifying the stratigraphy of the unconsolidated soil materials overlying bedrock.
2. Determine the depth to bedrock and obtain samples of the underlying bedrock.
3. Obtain additional stratigraphic information for comparative purposes by gamma logging.
4. Obtain representative ground water samples for contamination field screening (by headspace GC analysis) at specified depths within the saturated soil column.

The selection of the appropriate drilling method to meet these four objectives has been made with consideration of the following constraints:

1. The drilling method must permit the collection of representative ground water samples at specified depth intervals for field laboratory screening for selected compounds. Therefore, the method must NOT introduce foreign liquids into the soil near the cutting edge of the drill stem, and must provide sufficient working area within the drill casing to lower a small pump for purging ground water or a bailer or similar device for the collection of a representative ground water sample.
2. The method must be capable of installing temporary casing to isolate separate aquifers if encountered during the drilling process. To be cost and time efficient, it should not be necessary to change drilling rigs or get involved in long changeover times with one rig to install this temporary casing. Implied in this constraint is the ability of the drilling method to continue to drill through the temporary casing.
3. Upon reaching bedrock in the boring, the method must be capable of obtaining a core sample at least ten (10) feet in length of the bedrock. Again, cost and time efficiencies demand that one rig with a minimal changeover time be used.
4. Since there exists some possibility for encountering contaminated soils during drilling, the method must control any solids and fluids removed from the boring.

After long consideration and evaluation of various drilling methods, and other options for the collection of subsurface samples, a procedure for drilling these borings has been identified. This procedure meets all of the defined objectives and operates within the constraints imposed by those objectives.

The selected method is an air-rotary technique known as the Dual-Tube Reverse Circulation Method. This method consists of a water-tight steel casing or outer tube of approximately 4.5 inches in diameter. Within this outer tube there is an inner tube of 2.4 inches internal diameter. This inner tube is connected to the outer tube at specified intervals, leaving an annular space between the dual tubes. The lead casing of the inner tubes uses either a tri-cone rotary bit or an open-faced toothed bit for cutting soil materials. The relative positions of the two tubes are such that the leading edge of the bit is at the same depth or lags slightly behind the leading edge of the outer tube. High velocity compressed air, which is injected down the annulus formed by the two tubes and through the bit, carries cuttings to the surface via the inner tube. Cuttings are conveyed to a cyclone where the air velocity is reduced, the air is discharged out the top and the solids (soils) fall out the bottom. The high velocity of the air stream returns cuttings from depth to the surface with little discernable delay. When it is necessary to provide permanent casing within a boring to either isolate formations or to provide a cavity within which a monitoring well can be constructed, the dual-tube method requires the use of a separate casing, known as the overshot pipe.

The overshot pipe is steel with an inside diameter of approximately five (5) inches. To install the overshot pipe, high pressure water and/or air and hydraulic pressure are used to jet the overshot around the dual-tube. At some sites, it can be anticipated that a confining layer separating water bearing units will be found at some depth. If contamination exists in the overlying water-bearing unit, it will be necessary to isolate the upper portion of the boring from the contamination so that contamination cannot migrate through the boring to the lower water-bearing unit as drilling proceeds. This would be accomplished by first using the dual-tube to drill to the confining layer. The overshot pipe would be advanced to the depth of the confining layer and a seal would be made. Dual-tube methods would then be used to continue the boring through the confining layer and into the underlying formation(s).

The dual-tube string, as mentioned above, has an inside diameter of 2.4 inches. This cavity is continuous from the top of casing through the bit if the open-faced toothed bit is used as the cutting tool. If coring of bedrock is necessary, it can be accomplished through the dual-tube string (with open-faced bit) using AQ coring equipment. AQ coring equipment has an outside diameter of 1.89 inches and yields a core with an outside diameter of 1.062 inches.

This description of the dual-tube method and equipment makes it clear as to why it is the selected method for the soil borings. The dual-tube method provides continuous soil samples (although not for headspace screening purposes), formation isolation capabilities and bedrock coring all from the same drilling rig. Other, less obvious advantages can also be found in the dual-tube method. These include:

1. Air discharge from the cyclone, if contaminated, can be directed well away from the work area with the use of a hose connected to the top of the cyclone.
2. Soil discharge from the cyclone can be directed directly into a container, thereby controlling contamination (when present), and keeping the work area clean.
3. Representative ground water samples can be collected from within the dual-tube.
4. No foreign liquids are used during drilling.
5. The method is very fast; at other locations in similar soils, 100-foot soil borings have been completed in a period of three hours or less.

The disadvantages of the dual-tube method are no more restrictive than any other drilling method. Heaving liquified soils are always a problem when they enter the drill casing, and no drilling method can deal with them well. The only time soil heave is likely to occur is when the air-flow is shut off to allow sampling of ground water. Soil heave in this situation is not a problem provided that it is recognized that the first cuttings returning when the air-flow is resumed are those that have heaved into the casing. Another potential disadvantage of using the dual-tube as described above involves the need for the open-faced drill bit when bedrock coring will be required. Boulders approximately one-foot in diameter can "seat" themselves directly on the bit, making it virtually impossible to drill through. The tri-cone rotary bit will be used on all borings that will not be coring bedrock.

**SAMPLING
WITH THE
DUAL-TUBE METHOD**

Ground Water Sampling by Dual Tube Drilling

Using this method eliminates cross-contamination

By Lori Huntoon Pencak

One of the most challenging tasks facing the ground water professional is evaluating the vertical distribution of subsurface contamination. Dual tube drilling methods allow the collection of ground water samples with depth during drilling, and minimize cross-contamination while providing site-specific information within the borehole.

Collecting ground water samples with depth is useful not only in detecting the contamination, but also as a design tool for monitoring systems. Multiple monitoring nests are often installed in the initial phase of a hydrogeologic investigation in an attempt to evaluate the distribution of vertical contamination.

A common problem which arises is the improper vertical placement of screened intervals, due to lack of existing site information. One approach to designing a monitor well network is the collection of ground water samples with depth during well installation.

The use of screened hollow stem augers has proven to be very use-

ful in this type of ground water sampling. However, the utility of the dual tube drilling method is quickly being understood as an extremely efficient and cost-effective method for determining specific aquifer information from the collection of ground water samples with depth.

Dual Tube Methods

Dual tube drilling, also called reverse circulation drilling and center sampling recovery, provides the collection of continuous representative samples from a borehole with virtually no cross-contamination of the samples. In addition to the collection of geological samples, ground water samples also can be collected from the borehole during drilling operations in order to determine differences in contaminant concentrations with depth.

This is possible due to the unique design of the drilling system. Dual tube drilling uses rotary drilling methods, including either a rotary bit or down-the-hole hammer to advance through the formation. The flush-threaded, dual-walled

SOURCE: DRILL SYSTEMS INC.

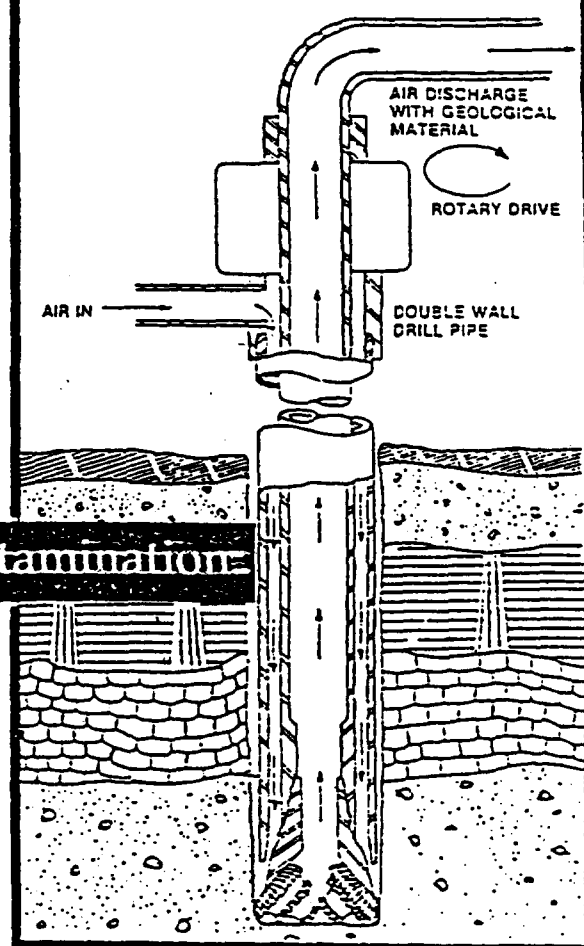


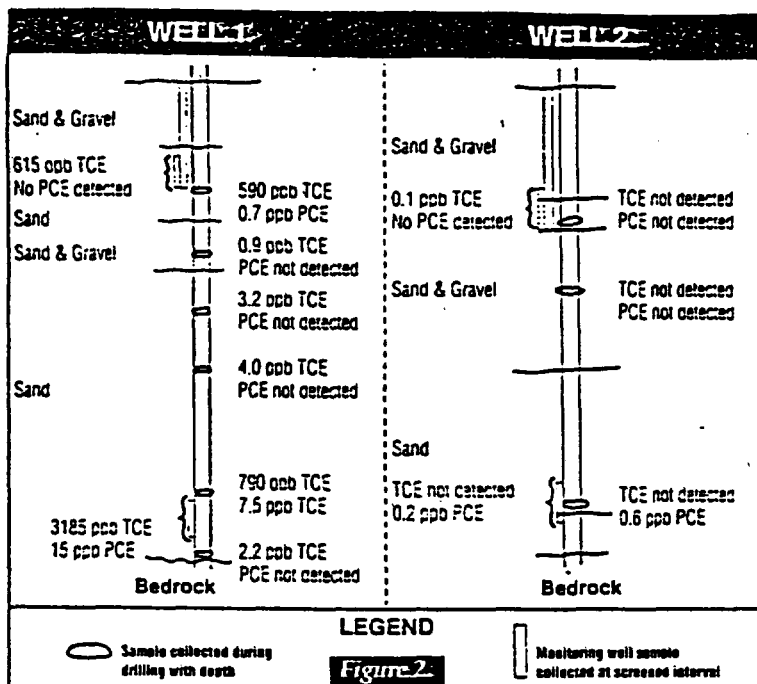
Figure 1. In reverse circulation sampling, air is injected down the outer annulus of dual-walled pipe, causing air and samples to flow up the inner pipe.

pipe is joined to the bit by a drill bit sub, and both are advanced simultaneously (see figure 1).

The drilling fluid, preferably air or mist, is forced down the outer annulus of the dual-walled pipe, and directed to the center of the pipe. The size of the borehole is such that there is a minimal clearance between the bit and the drilling pipe, which minimizes cross-contamination along the annulus of the borehole.

The cuttings are discharged through the inner annulus to the surface within fractions of a second, allowing immediate determination of the lithology. It also is important to note that formation

(Continued on next page)



cuttings and ground water samples don't come into contact with the overlying borehole materials.

During drilling, ground water samples can be collected at selected intervals as the borehole is advanced. Due to the design of the system, ground water is allowed to enter only through the open bottom of the borehole. Thus, the sample collected is representative of the actual ground water in the formation at the open interval.

Samples can be collected by lowering a pump intake line or a bailer through the inner tube of the drilling column. Several volumes of water may be removed prior to

drilling tools — rather, place the line several feet above the bottom.

If possible, use a photoionization meter during drilling and sampling procedures, and screen the samples prior to submitting them to the laboratory. It's helpful to the lab if you can estimate whether the sample will have high or low detects of contaminants. High levels of contaminants when not expected can result in laboratory equipment being shut down for days in order to decontaminate the machines.

Samples can be collected in VOA vials and immediately analyzed by gas chromatography

collection in order to insure a representative sample.

When using a bailer, lower and raise the bailer slowly with a smooth motion to avoid volatilization of potential contaminants. When using a pump, don't allow the intake line to extend to the bottom of the

methods in the field, and/or transported to a laboratory for analysis. It's best to collect at least two vials for each sample, to allow for sample breakage and unexpected analytical duplicates. Additional samples can be collected for field screening, indicator parameters (pH, specific conductance, temperature) and additional laboratory analyses.

Properly label the sample bottles with the well number, sample depth, date and time. Samples should be immediately stored on ice and warmed to room temperature prior to analysis.

During the collection of ground water samples, stringent decontamination procedures should be followed. These include:

- Washing the sampling equipment with laboratory-grade detergent and several rinses with distilled water between each sample.
- Replacing the rope or tubing after each sample collection.
- Using gloves when handling the sampling equipment or bottles.

It's important to note that in fine-grained formations, bridging of sand in the drilling tools may not allow the collection of a ground water sample.

Case Study

A subsurface investigation was completed to determine the source

Do's and Dont's for Collecting Samples When Using Dual Tube Drilling

- DO decontaminate the sampling equipment thoroughly between each sample.
- DO replace the bailer rope or plastic tubing between each sample.
- DON'T surge the bailer up and down. Lower and raise the bailer into the well slowly with a smooth motion to ensure a representative sample and to avoid volatilization

of potential contaminants.

- DO collect at least two vials for each sample to allow for sample bottle breakage and unexpected analytical duplicates.
- DO use a field ionization meter to screen the samples before submitting them to the laboratory. It will help if you can estimate if the sample will have high or low detects of contaminants.

- DO label the samples immediately after collection. Place the samples on ice, complete the necessary chain of custody records and transport the samples to the laboratory as soon as possible.
- DO use cross-section maps in addition to tables to evaluate the results received from the use of vertical sampling with depth during drilling. □

and extent of a trichloroethylene (TCE) ground water contaminant plume. Several deep monitoring wells were installed at the site, using the dual tube method.

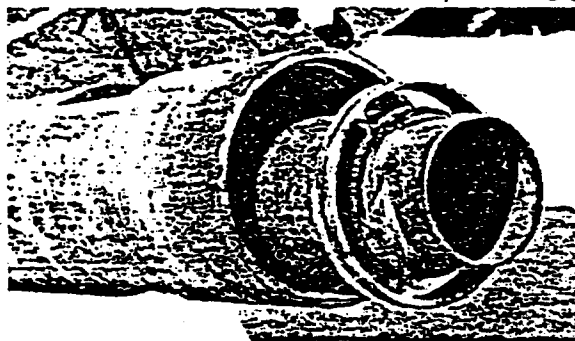
Water quality samples were collected at 20 foot intervals to a depth of 150 feet in order to determine the variations in ground water quality with depth.

Concentrations of contaminants were detected at two distinct depths within the aquifer, which may have been missed during the installation of a standard monitoring network due to improper vertical placement of screened intervals. Figure 2 shows the results of the samples which were collected from two of these wells during drilling.

The contaminant plumes detected during the investigation may have been missed during the installation of a standard monitoring network due to improper placement of screened intervals. Figure 3 shows the hypothetical place-

ment of a screen in coarse material overlying bedrock.

However, in this case, the contaminant plume wasn't located directly above the bedrock, but rather tens of feet above it. The installation of a screen in the coarse material would indicate a lack of contamination (as shown in the



An end section of reverse circulation drill pipe.

collection of the sample at depth in figure 2).

In addition, concentrations of the contaminant in between these plume areas were minimal or nonexistent, insuring that cross-contamination between the sam-

ples was minimized.

Evaluating the vertical distribution of contamination with depth is critical in determining the appropriate screen depths of monitoring wells. The collection and analysis of ground water samples with selected depths during drilling provides the water professional with a definition of ground water quality with vertical distance.

And the most efficient and cost-effective method of collecting ground water samples with depth is through the dual tube drilling method, which allows collection of the samples at selected depths while minimizing the chance of cross-contamination. □

Lori Huntoon Pencak is staff hydrogeologist — project development for Geraghty & Miller Inc., Milwaukee, WI.

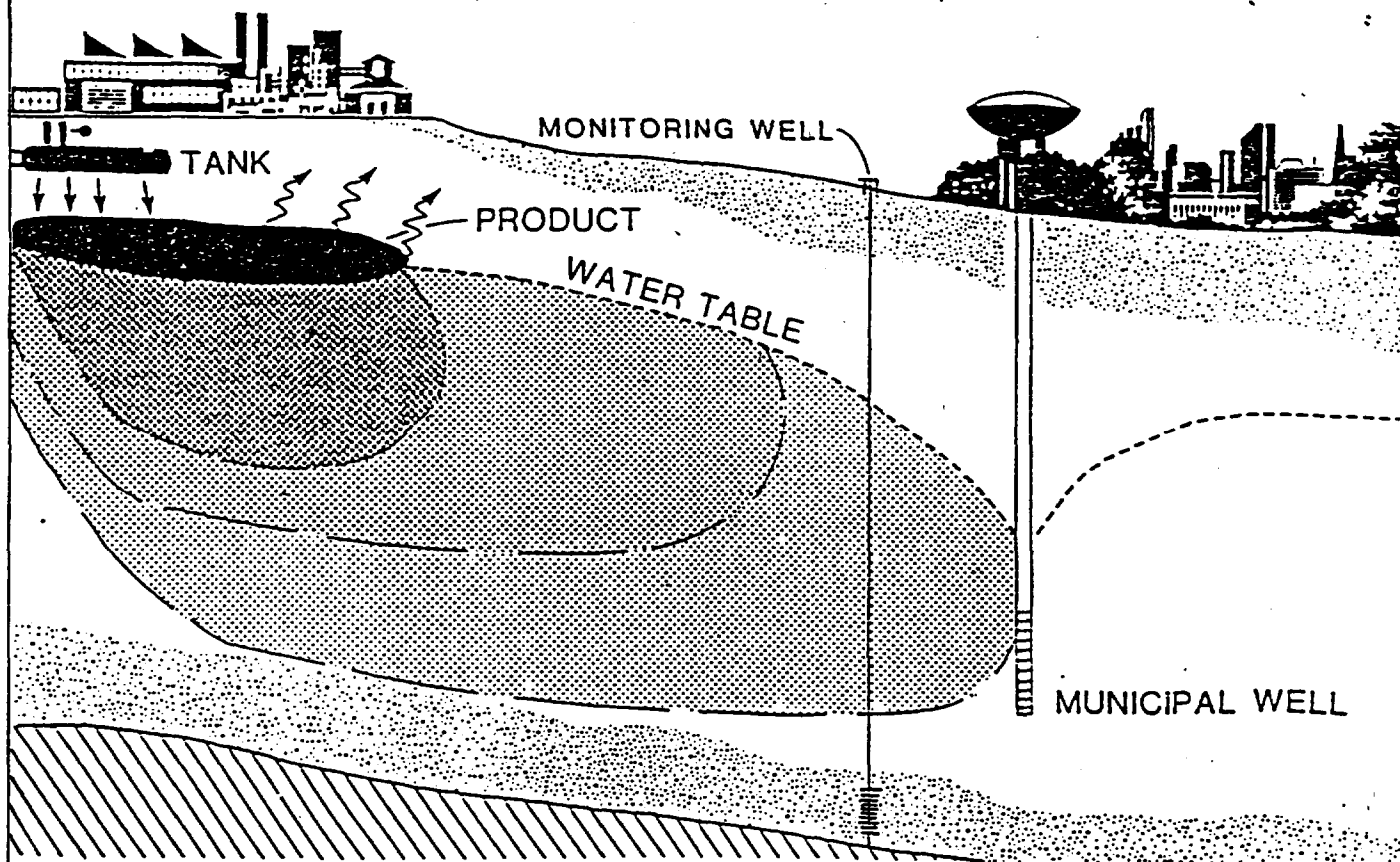


Figure 3. Specific placement of monitoring wells is necessary to determine the extent of a contaminant plume.

Appendix A-A3

Operator's Manual for the OMNI IV Magnetometer



OPERATION MANUAL

OMNI IV

TIE-LINE MAGNETOMETER

PPX-402

Printed in Canada

30.06.85

WARRANTY

EDA Instruments Inc. warrants that at the time of shipment the goods shall be free from defect in material and workmanship. Notice of any defect must be given to EDA Instruments Inc. in writing immediately upon discovery of any such defect and must be within the time of the warranty stated in the face hereof or if none stated within 1 year from the date of shipment of the goods by the purchaser. EDA Instruments Inc. obligations under this warranty are limited to the repair or replacement of the parts or materials which upon examination by EDA Instruments Inc. are established to be defective within this warranty and in no event shall the corporation's obligations under this warranty exceed the cost of the goods sold. For the purposes of repairing or replacing parts or material under this warranty the purchaser shall forward at its own risk and freight prepaid the goods to EDA Instruments Inc. This warranty ceases to apply to any goods altered or repaired by anyone other than EDA Instruments Inc or its authorized representative.

NOTE: EDA Instruments Inc., reserves the right to revise specifications in order to improve upon product performance without written notice.

WARRANTY SERVICE

When warranty service is required or when technical consultation is required, contact:

In Canada and Internationally: In U.S.A.:

EDA INSTRUMENTS INC.,
1 Thorncliffe Park Drive
TORONTO, ONTARIO
CANADA, M4H 1G9
Telephone: (416) 425-7800
TELEX: 06-23222 EDA TOR

EDA INSTRUMENTS INC.,
5151 Ward Road
Wheat Ridge (Denver)
Colorado 80033
Telephone: (303) 422 9112

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WARNING

When the instrument requires servicing by the customer, EDA Instruments Inc., suggests that the service department of EDA be contacted first, because instrument withdrawal from the case may cause damage.

IMPORTANT WARRANTY NOTE

EDA Instruments Inc., reserves the right to null and void the warranty if and when it can be determined by specially trained personnel or an authorized agent that the instrument has been misused.

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SECTION 1

GENERAL INFORMATION

1.1 SCOPE OF MANUAL

This manual describes the OMNI IV Tie-Line Magnetometer designed and manufactured by EDA Instruments Inc., Toronto, Canada. The information is provided for personnel responsible for operation of the unit and should be read prior to operation and testing to obtain a full understanding of all of the features incorporated. Technical Specifications are given in Table 1-1.

1.2 PURPOSE OF INSTRUMENT

The OMNI IV is a portable, microprocessor-based total field magnetometer system which is capable of correcting its own field survey data for diurnal variations without the need of a base station. A measurement can be read and stored in only three seconds. The data is highly repeatable. A second reading is not usually required.

The OMNI IV is a multi-purpose instrument designed to operate in four different functions or modes:

- a. Tie-line magnetometer (3).
- b. Portable field magnetometer (3).
- c. Recording base station magnetometer (4).
- d. Gradiometer (5).

Software. The instrument function (mode) is predetermined by the software that is resident in EPROMs inside the instrument. The software cannot be changed by the operator. The level of the software installed by the manufacturer is identifiable on the instrument readout display as part of the serial number. The procedure for obtaining the software level display is described in the operation section. The software level identification number is shown in () in a. through d. above. Software with a higher identification number also includes the software for the lower numbers.

The primary purpose of this microprocessor-based instrument is to:

- measure and store the total earth's magnetic field;
- identify the location of that measurement by recording either the grid coordinates or a record number;
- record the time that the measurement was made.

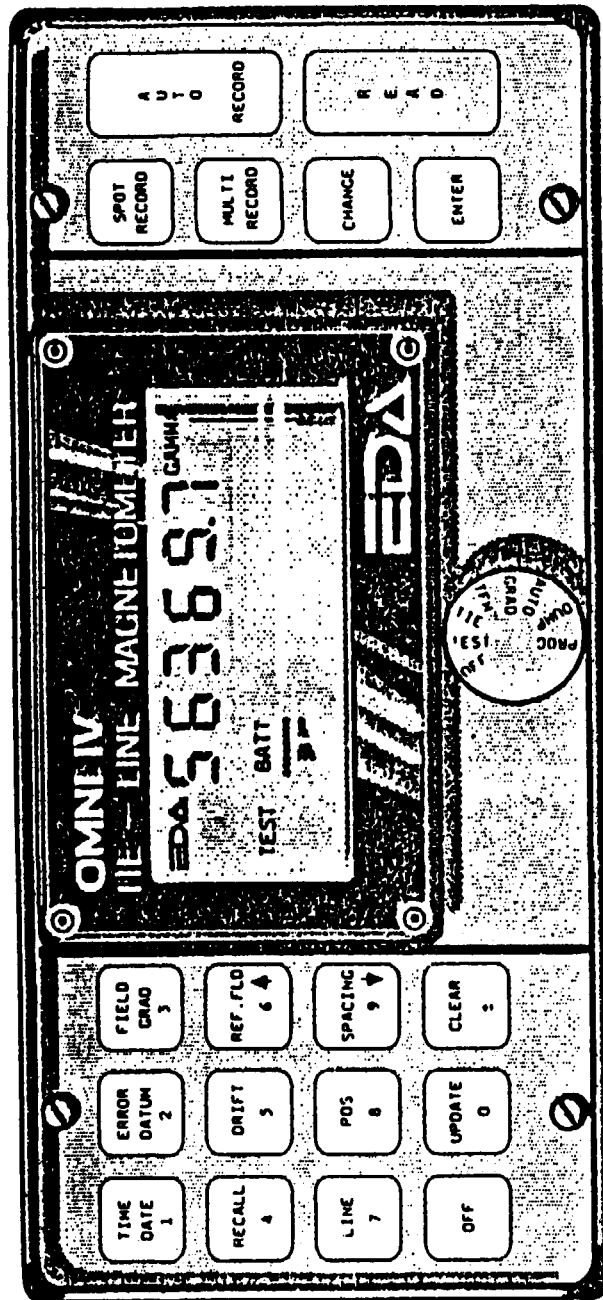


Figure 1-1 OMNI IV Tie-Line Magnetometer

In addition, the OMNI IV can correct its own field measurements for diurnal variations without the need of a base station, by utilizing either the tie-line or looping point method of survey. However, the instrument stores only the raw data; it does not store corrected data. Corrected data is fed to the instrument output connector during the data dump mode for recording in a peripheral computer or printer. The raw data is retained until the instrument is re-initialized even after corrected data has been computed.

Data stored in memory is completely protected by a lithium battery. This battery also powers the real-time clock.

Field Measurement Accuracy. The following design features eliminate the need for taking multiple readings of poor instrument performance:

- Patented signal processing technique.
- Constant energy polarization that maintains equal energy to the sensor for each measurement.
- Processing sensitivity to within ± 0.02 gamma.
- Automatic fine tuning which uses the previous reading as the basis for the next reading.

Choice of Function. The choices are categorized in four functions associated with the resident software. However in each category, there are also numerous feature choices, based on operator preference.

TIE-LINE MAGNETOMETER. In this mode, the OMNI IV is able to store raw data obtained using either the looping survey or tie-line survey method. This reference data is stored in a separate memory at the beginning of each survey. Then total field readings are taken and stored in another memory together with the repeat field readings at the tie-points. At the end of each survey day, the data in the two memories are merged so that the total field data is corrected for diurnal variations automatically. These corrected values are outputted from the instrument immediately (ie, not stored).

In the tie-line mode, the OMNI IV can store looping or tie-line data in any one of the following ways:

- a. Using one base point for reference in the looping survey method.
- b. Using one tie line consisting of a number of tie-points for reference in the tie-line survey method.
- c. Using multiple tie lines for reference in the tie-line survey method.
- d. Using a random scattering of tie-points as reference.

The flexibility offered allows the operator to choose the most appropriate tie-line method best suited for the survey, depending upon such factors as the size and character of the survey grid. For smaller survey areas the looping method is practical but for larger areas, tie-line references become necessary. The tie-line algorithms are based on a linear diurnal variation between adjacent tie points. Therefore the accuracy of the corrected data is a function of the time lapse between measurements at adjacent tie-points and any erratic behaviour of the diurnal variation during that same period. Typically, the time lapse should be less than 30 minutes. Using the multiple tie-line feature, the requirement for base station magnetometer recordings can be eliminated from some surveys.

Up to 100 tie points can be stored in one survey area. Alternatively, these 100 tie points can be divided into extensions of survey areas as needed. The tie point and tie-line measurements are stored for the duration of the survey. Tie-line data can be recalled even if stored on different days. However, raw field data can only be entered daily during a midnight-to-midnight period.

The OMNI IV can also calculate the drift between established tie points so that variations in the earth's magnetic field can be seen readily. Similarly, the OMNI IV can also calculate the desired diurnal drift measured between consecutive tie point readings.

In the OMNI IV software, there is a programmable datum. A datum (eg, 57,000 γ) can be entered into the instrument. Then, when a reading is taken, this datum, which is a coarse background value, is subtracted from the reading. This provides a small value of the differences to facilitate plotting and interpretation of data.

The tie-line capability is also applicable when the instrument is used as a gradiometer. The operator can therefore obtain 'corrected' total field data using the linear diurnal variation algorithm without relating to base station magnetometer readings.

PORTABLE FIELD MAGNETOMETER. In this mode, the OMNI IV measures and stores the total field magnitude; time of measurement; grid coordinates; and direction of travel in the survey grid. It also computes and stores the statistical reading error, the signal strength and the rate of decay. The memory can store up to 1400 data blocks thereby eliminating the need to record data manually. Accumulated data can be dumped to printers, to magnetic cassette recorders or in parallel with a base station magnetometer to a computer.

The operator has a choice of three data storage modes:

- Spot record. Each reading is assigned a record number automatically. This can be used to identify readings which are taken at locations which do not correspond to survey grid coordinates.
- Multi record. More than one reading can be taken at a survey grid coordinate without updating the current station or line number.
- Auto record. These readings are taken automatically based on a preprogrammed time interval.

The direction of travel is recorded using the primary cardinal points (N, S, E, W) and by increments in the position indication.

The statistical error is calculated for each measurement based on an internal algorithm. The result of the calculation is displayed to provide the operator with an accuracy factor. From this it may be determined whether or not an additional reading will be required.

Similar to the tie-line mode, the following features can be selected:

- If the last reading is unwanted, it can be cleared.
- The difference between the current reading and previous reading is calculated automatically and can be displayed upon push-button command.
- A preprogrammed datum or coarse magnetic field value can be subtracted from each field data measurement to simplify plotting of field survey results.
- Diurnal corrections are calculated automatically.

BASE STATION MAGNETOMETER. In the base station configuration, the OMNI IV measures and stores in its memory the diurnal variations in the earth's magnetic field. The instrument is designed specifically to meet base station specifications for airborne and ground surveys. Readings are taken automatically based on an interval which is programmed by the operator. The interval can be varied from 5 seconds to 60 minutes in 1 second increments. The memory has a capacity of 5000 data blocks. This provides a 7 hour capacity at the 5 second frequency.

The data that is being stored can be outputted simultaneously in a digital or ASCII format to a choice of data collection systems.

When the base station is connected in parallel with the compatible field magnetometer, it can correct and dump the total field measurements. The corrections made are for diurnal variations and reference field values.

The magnetic field activity is recorded in the following format:

- Time of measurement.
- Total field magnitude.
- Difference from the reference field value.
- Difference from the previous reading.
- Sequential record number.

The OMNI IV can also be programmed with a reference field value (eg, 57,000 γ). This 'bulk' value is then subtracted from each reading so that only a small but significant value (which is the variation between readings) is recorded.

GRADIOMETER. When the OMNI IV is programmed for use as a gradiometer, a dual sensor staff is connected. The sensors are incorporated in a staff with the sensors spaced vertically 0.5 m apart. The OMNI IV reads and stores the measurements of both sensors simultaneously, thereby providing a true measure of the total field and the gradient of the total field. Simultaneous readings remove the effects of diurnal variations and magnetic storm interferences from the data.

The stored data includes:

- The gradient of the total field.
- The total magnetic field measured by the upper sensor.
- The time of measurement.
- The survey grid coordinates where the measurement was made.
- The statistical error of total field reading made by the lower sensor.
- The signal strength and decay rate measurement of the lower sensor.

Using the tie-line method, or in conjunction with an OMNI IV base station, the total field measurements made by the upper sensor can be corrected for diurnal variation. Because the sensor readings are simultaneous, diurnal magnetic variations of the gradient measurements are cancelled.

In the gradient mode, the OMNI IV has improved resolution of total field anomalies. Therefore it defines more sharply the magnetic responses determined by total field data. Closely spaced anomalies are delineated individually rather than being identified collectively as one broad magnetic response. There is also direct delineation of vertical contacts.

Therefore, the OMNI IV is an ideal contact mapping tool especially in vertical or near vertical contacts or fault zones. These vertical contacts are depicted at the zero line of gradient contours or profile values. Vertical bodies analogous to a dyke can also be mapped effectively. The OMNI IV is also effective in enhancing anomalies near the surface. Any shallow source exhibits a higher frequency anomaly than a deeper low-frequency anomaly. This comparison can provide an on-the-spot approximation of the depth of the anomalous source. The capability of the OMNI IV to differentiate between higher and lower frequency responses removes background regional gradients from anomalous residual responses.

The OMNI IV can be used in a combined gradient and base station operation. It can be programmed to cycle automatically every 5 seconds in the gradient mode. This feature can be used in stationary or mobile applications.

The OMNI IV sensors are mounted in a sectional aluminum staff in which sections can be added or subtracted. Similarly, there are optional separations of the sensors of 0.5 m or 1.0 m. These can be used to obtain interpretive information especially for surface anomalous conditions (eg, determining if the field is linear or curved).

1.3 FUNCTIONAL DESCRIPTION

The OMNI IV Tie-Line Magnetometer is a ruggedized compact portable instrument designed specifically for field operation. The instrument is extremely accurate and flexible. It contains a microprocessor and associated circuitry (CPU) for monitoring, storing and processing data. Basically, the OMNI IV has two memories: One for storing data taken at a base reference point and along a tie line; the other for storing field measurements of the actual survey.

The actual survey points would comprise an imaginary grid covering the survey area. In the tie-line application, one or more of those grid lines (or even selected grid points) are established as the tie line (or tie point) references. Measurements of the earth's magnetic field are made, usually at the beginning of a survey, at each selected tie point. Then, field measurements are made at the survey grid intersection points including subsequent (ie, repeat) readings at the tie-line points. Variations in the readings taken at a tie-line point provides a reading of the diurnal variation experienced at that point. These are used as the basis for computing the corrections for readings at adjacent survey points. In this computation for correction of readings, the variation between two readings at a tie point (ie, the diurnal variation) is compared with the variation in the two readings at the previous tie point. The times that these variations were recorded are also recorded. Therefore, the diurnal variation associated with any intermediate survey point between two tie points can be computed with extreme accuracy confidence and the correction can be applied to the reading taken. This computation is carried out within the instrument automatically without operator assistance.

The algorithm assumes that the diurnal variation is linear with respect to time. This is usually valid if the interval between two readings at the same point is short (ie, less than 30 minutes). It may be less accurate during periods of increased activity. For critical measurements, the base station method provides greater accuracy due to the time interval factor; however, the inaccuracy is a function of the distance from the base station to the point of measurement.

The instrument performs all of the following functions initiated by pressure-sensitive push-button commands:

- a. It provides a visual readout of the total magnetic field amplitude. When the instrument is used as a gradiometer, it also provides a visual readout of the gradient of the total field.
- b. It provides a visual readout of the time and date of a measurement upon push-button command.

- c. It provides a visual readout of the statistical error associated with the total field measurement upon push-button command.

- d. It assigns the grid coordinates of a measurement in terms of line and position numbers in the survey grid.

- e. It provides incremental update of the position and line numbers along a line including changes to the line and position spacing increments at any time.

- f. It provides the drift or difference in gammas between the previous and current field reading.

- g. It records all of the parameters, a through g, in memory by selecting one of three record keys.

- h. It assigns a record number to each total magnetic field measurement. This can be used instead of grid coordinates.

Physical Dimensions		Wt(kg): w x h x d(mm)
Instrument console only	2.8:	238 x 150 x 250
Battery belt	1.2:	540 x 100 x 40
(Alkaline)		
Battery cartridge	1.8:	235 x 105 x 90
(Lead acid)		
Battery belt	1.8:	540 x 100 x 40
(Lead acid)		
Sensors		
Remote sensor	1.2:	56 dia x 220
Gradient sensor		
0.5 m separation (standard) ..	2.1:	56 dia x 790
1.0 m separation (optional) ...	2.2:	56 dia x 1290
Environment		
Electronics		
Operating temperature range ... -40°C to +55°C		
Relative humidity 0 to 100% (weather-proof)		
Sensor		
Temperature range -45°C to +55°C		
Relative humidity 0 to 100% (weather-proof)		
Standard Memory Capacity		
Total field or gradient 1400 data blocks or sets of readings		
Tie-line points 100 data blocks or sets of readings		
Base station 5000 data blocks or sets of readings		
Electronics		
RS-232C serial I/O 2400 baud; 8 data bits; 2 stop bits; no parity		
Electronic console Enclosure contains electronics and battery pack (If not contained in separate belt). Front panel includes liquid crystal display (LCD), keypad and MODE selector.		
Power supply Internal battery pack or external battery belt; or +12 V car battery if used as a base station.		

Table 1-1 Technical Summary

Specifications	
Dynamic range	18,000 to 110,000 γ . Roll-over display suppresses first significant digit upon exceeding 100,000 γ .
Processing sensitivity	$\pm 0.02 \gamma$
(total field)	
Statistical error resolution	0.01 γ
Absolute accuracy	$\pm 1 \gamma$ at 50,000 γ at 23°C. $\pm 2 \gamma$ over total temperature range.
Display resolution (total field)	0.1 γ
Tuning method	Microprocessor calculates precise tuning. Value utilizing a specially-developed timing algorithm.
Automatic fine tuning	$\pm 15\%$ relative to ambient field of last stored total field value.
Tracking range	18,000 to 110,000 γ
Gradient tolerance	6,000 γ per metre (field proven)
Tuning indicator	Descriptor on display (TUNE) commences to flash on and off. Audio alarm activated also. (New field value must be entered into system.)
Display	Custom designed, ruggedized liquid crystal display with an operating temperature range of -40°C to +55°C at 100% RH. The display contains six numeric digits, two decimal points, battery monitor, signal decay rate and signal amplitude monitor and function descriptors.

Table 1-1 Technical Summary (Cont)

SECTION 2

PHYSICAL DESCRIPTION

2.1 SYSTEM COMPLEMENT

The instrument can be assembled in three configurations, with variations depending upon the type of sensor selected, the preferred power package and peripherals.

The configuration complements are given in Table 2-1.

STANDARD SYSTEM (Software Code 3). The basic configuration is for use in total field, tie-line and looping applications. It comprises:

- a. A console which is carried on the chest and operated in that position. (The console includes a 1400 data block memory for field magnetometer applications.)
- b. A shoulder harness to support the console comfortably so that both hands are free.
- c. A remote sensor which is complete with a 3 m cable for attachment to the console.
- d. A sectionalized pole assembly (four, 60 cm sections) which supports the sensor.
- e. A rechargeable battery cartridge.
- f. This system also includes a plug-in battery charger for use with 110 Vac or 220 Vac circuits, and an operation manual.

BASE STATION CAPABILITY (Software Code 4). This option comprises a standard system plus the following:

- a. A standard console with a 1400 data block memory for field magnetometer applications and an enhancement of a 5000 data block memory for base station applications.
- b. A 28 m base station cable extension so that the sensor can be located remote from the console (eg, console located in a shelter).
- c. A rope joiner. This joiner provides attachment points for securing the sensor and pole upright using non-metallic guy lines.

BASE STATION AND GRADIENT CAPABILITIES (Software Code 5). This option is the same as the standard configuration except for the following enhancements:

a. A standard configuration with a 1400 data block memory for field readings and an enhancement of a 5000 data block memory when in the base station mode.

b. The remote sensor is replaced by a dual sensor with the sensors spaced 0.5 m apart. Optionally, the sensors can be spaced 1.0 m apart.

System Peripherals and Accessories. Each of the configurations can be used with the peripherals listed in Table 2-1. An optional transit case is also available as an accessory for the console, sensor, sensor pole, battery pack and battery charger.

2.2 COMPONENT DESCRIPTION

Standard Instrument with Remote Sensor. The electronics console is carried on the chest with the instrument panel facing upwards. The console is supported in a dual shoulder-type harness. The sensor is mounted on a sectional pole, 2.5 m long and connected to the console through a flexible cable. It is hand carried similar to a staff.

Instrument with Gradient Sensor. The electronics console is carried on the chest with the instrument panel facing upwards. The console is supported in a dual shoulder-type harness. The gradient sensor comprises two sensors mounted vertically on the hand-held pole and separated by a distance of 0.5 m (standard) or 1 m (optional). This permits simultaneous measurements from each sensor.

INSTRUMENTS CONSOLE. The primary electronics, data acquisition circuit, microprocessor and memories are built into a rectangular, aluminum, weather-proof case with the instrument panel facing upwards.

Display. Data and information is displayed on a custom-designed, ruggedized, liquid crystal display (LCD) which operates in temperatures ranging from -40°C to $+55^{\circ}\text{C}$. The display includes a six-numeric digit readout, decimal point, battery status monitor, signal decay rate and signal amplitude monitor, and function descriptors. The internal heater is activated automatically at -25°C during the survey. Make sure that the console MODE selector is set to OFF overnight if the temperature is expected to be below -25°C . The OFF selection turns off the display heater.

Operator Keys. The operator keys are grouped into two keypads located on each side of the LCD. The 12 keys on the left hand side are for programming the instrument. The 6 keys on the right hand side are for taking measurements and recording them. The key functions are described in Section 3.

	Standard OMNI IV	OMNI IV with Base Station Capability	OMNI IV with Base Station & Gradiometer Capabilities
OMNI IV Console	✓	✓	✓
Memory (1400 data blocks)	✓	✓	✓
Memory (5000 data blocks)		✓	✓
Remote Sensor PPX-326R	✓	✓	
Gradient Sensor (0.5 m separation) PPX-528			✓
Gradient Sensor (1.0 m separation) PPX-			Optional
Pole Assembly (4 x 60 cm sections) PPX-360	✓	✓	✓
Cable Extension, 28 m PPX-436		Optional	
Rope Joiner (guy rope attachment) PPX-457		✓	
Battery Belt PPX-	✓	✓	
Battery Cartridge (rechargeable, sealed, lead acid) PPX-365	Optional	Optional	✓
Battery Charger 110/220 Vac PPX-255	Optional	Optional	✓
Operation Manual PPX-402	✓	✓	✓

Table 2-1 OMNI IV System Complements

Options and Accessories:

Thermal Printer (AC/DC)

40 character
DCU-400

Thermal Printer (AC only)

40 character
DCU-040

Dot Matrix Printer (AC only)

80 character
DCU-080

Paper, Thermal Printer

DCX-441

Paper, 80 column fan fold

DCX-445

Communications Interface Adapter

DCA-100

Interconnect cable (data dump) for single OMNI IV

DCX-442

Interconnect cable (data correction to TRS80/100), male (1-020-00335)

Interconnect cable (data correction to printer), male DCX-443

Interconnect cable (data correction to Heathkit H-14 printer), female (1-020-00330)

Interconnect cable (data correction to HP-85 option 0), male DCX-446 (1-020-00335) Note: Female RS-232 serial interface connector.

Interconnect cable (data correction to HP-85 option 1), female DCX-446 (1-020-00330) Note: Male RS-232 serial interface connector.

Interconnect cable (data correction to IBM PC), female DCX-447 (1-020-00330)

Cable Connectors. There are two cable connectors located on the rear of the instrument (ie, remote from the control panel). When the console is being used (ie, chest mounted):

- The one on the operator's left side connects to the sensor.
- The one on the operator's right side is for data dumping. It is an RS-232C serial input-output connector. The interface parameters are: 2400 baud; 8 data bits; 2 stop bits; no parity.

REMOTE SENSOR. The sensor consists of two helical coils of copper wire connected in series in a noise-cancelling mode with at least 50 dB attenuation of external noise. The coils are immersed in a hydrocarbon-rich liquid inside a light-weight, leak-proof cylinder. The sensor cylinder is mounted inside a thin-wall fiberglass tube. The coils are positioned with their axes parallel to each other. The interconnections are carried through a cable, 3 m long and terminated in a connector which interfaces with a connector on the rear of the OMNI IV.

Remote Sensor Pole. This sensor pole consists of a sensing head mounted on a pole. The pole consists of four short rods which engage end to end so that the sensor is approximately 2.5 m above the ground.

Dual Gradient Sensors. For the gradiometer application, two identical sensors are mounted vertically at the ends of a rigid fiberglass tube. In the standard configuration, the centres of the coils are spaced 0.5 m apart. An optional configuration separates the coils by 1.0 m. Interconnections are carried through a cable, 3 m long and terminated in a connector which interfaces with a connector on the rear of the OMNI IV. The sensor pole is the same as that used with the remote sensor.

Base Station Sensor. The base station application can use either a standard remote sensor or a dual gradient sensor. The interconnection cable between the sensor(s) and the OMNI IV can be 28 m long. This allows the instrument to be located in an enclosure while the sensor is located outside.

Base Station Sensor Pole. The sensor support pole is fitted with a rope joiner. This is a collar with anchor tie points for attaching three non-metallic guy ropes.

Battery Cartridge Power Supply. Three types of power supply are available (a. and b. are optional; C. is standard):

- A non-magnetic rechargeable battery pack with eight sealed lead acid cells.
- A non-magnetic rechargeable battery belt with eight sealed lead acid cells.
- An alkaline battery belt with 12 D size alkaline disposable power cells.

Table 2-1 OMNI IV System Complements (Cont)

Battery Belt. This is a webbed belt with a zip enclosure enclosed pouch. For the standard configuration, each system includes a battery belt for alkaline 'D' size batteries. Optionally, the belt comes with sealed, lead-acid rechargeable batteries. The batteries are placed in protective packing inside the pouch. Interconnections between batteries are made internally so that there are two outputs: One is the power output; the other is the battery charging and data dump connection. The power output cable is connected to a base plate which is similar in appearance to the battery pack which mounts on the base of the instrument. Therefore, when the base plate is attached to the instrument, the battery connections to the instrument are completed.

Battery Charger. Each system with the optional rechargeable batteries, includes a battery charger, MBC-100 (Part No. PPX-255) for use with the sealed lead acid battery pack. The batteries are recharged from a 110 Vac or 220 Vac source while the cartridge is located on the console, or in the belt.

Base Station Power Supply. In the base station mode, the OMNI IV can also be operated from an external 12 Vdc car battery.

NOTE

The characteristics of alkaline batteries require a program variation. For this reason, the second digit of the operator code is entered as a '9' (eg, OP39NN) for alkaline batteries and any other digit for rechargeable batteries.

Alkaline Batteries. The use of alkaline batteries is now possible. Previously, they could not be used because the unit sees a low battery voltage during polarization. Until now this was interpreted as sealed lead-acid batteries which require charging to prevent damage to the batteries. To bypass this protection monitor, it is necessary to enter an OP-code that indicates alkaline batteries (which have much lower voltage during load). Enter a '9' as the second digit of the code, ie, OP39NN for the total field unit; OP49NN for base station unit. The second digit of the operator code must be a digit other than 9 if normal rechargeable batteries are used. This prevents damage to the rechargeable batteries.

NOTE

Alkaline batteries cannot be used when the unit is operated as a gradiometer.

SECTION 3

PRINCIPLES OF OPERATION

3.1 INTRODUCTION

This section is confined to the principles of operation of the OMNI IV instrument. An explanation of the design concept and the derivation of constants used in the design concept are beyond the scope of this manual.

3.2 PRINCIPLES

The instrument includes a signal processing method which, for the first time, produces records which are meaningful statistically.

The instrument performs the following functions:

- a. It makes an accurate measurement of the total magnetic field strength and processes the information to verify its accuracy.
- b. It stores the measurement together with various parameters including signal strength and rate of decay.
- c. It records location data so that each measurement can be identified in terms of geographical coordinates (or by record number) related to a precise ground location.
- d. It records the date and time at which each reading was taken.
- e. It records the type of data recorded, whether it is corrected or uncorrected data, operator identification number, number of readings taken, and voltages of battery.
- f. It provides visual readouts of parameters based on keypad commands, ie, datum to be subtracted.
- g. It produces data block header information which identifies both the operator and instrument used.
- h. It provides three data output formats, for hard copy print-out and digital data transmission.
- j. It performs built-in test programs which provide a high confidence level in field measurements.

Circuit operations, computer programs and data logging requirements which fulfill these functions are described in the following paragraphs.

3.3 OPERATOR CONTROLS

Upper Panel. Visual readouts are provided on a custom designed liquid crystal display (LCD) which is capable of operating in ambient temperatures from +55°C to -40°C. The display utilizes a special low temperature fluid which allows the display to be operated down to -35°C. However, at this temperature, the response time is slow. A heater is built into the display. The heater circuit is controlled by a thermal relay so that it is turned on automatically when the ambient temperature is below -25°C. This is to ensure a specific response time. When the unit is turned off (ie, MODE selector to OFF), this heater circuit is disconnected to conserve battery power and the automatic temperature control is overridden. Therefore, in extreme temperatures, when the unit is not being used, it is recommended that it be stored inside, in the usual personnel accommodation environment to ensure that the display becomes operational with a minimum warm-up period.

Mode Selector. The instrument operating mode is established by the setting of the MODE selector. The measurements used in the self-correcting computations for survey measurements are recorded in the TIE mode. Survey measurements are made in the total field (TFM) mode. Survey gradient measurements, using dual (gradient) sensors, are made in the GRAD mode. Sequential measurements taken at the same time interval and at the same location, (ie, base station application), are made in the AUTO mode. Data block output occurs in the data dump (DUMP) mode. Data output in an analog profile plot format occurs in the program (PROG) mode. In the TEST mode, simulated field measurements are made and software diagnostics are performed. The OFF mode is used to power down the display heaters and electronics when the OMNI IV is not being used or during shipping. The memory and real-time clock is still intact in this mode.

Keypads. There are two keypads; the left hand pad is for programming the instrument; the right hand pad is for recording and modifying measurement and reference data. The keys are pressure sensitive push-buttons. The primary key functions which are parameter and command functions, are identified in black lettering (eg, TIME).

Note

The individual key functions are described in Part 4.

3.4 Battery Power

Field operations are carried out under a rigid computer-controlled program of conservation which maximizes the operational period between battery charging or replacement. This is accomplished during a brief, two second reading

sequence. Residual battery power is monitored for every reading. The OMNI IV also has a unique feature called 'constant energy polarization'. The same amount of power is used to polarize the sensor for each reading regardless of the state of depletion of the batteries. When the required amount of power is not available, a reading is prevented.

3.5 Reading Sequence

A reading sequence is started by operator command. This turns on the power and initiates the selected computer program. First, the computer is initialized, then the setting of the MODE selector is interpreted. If a total field measurement is being taken, the following programmed sequence occurs.

- a. Polarizing Cycle.
- b. Signal Analysis Cycle.
- c. Tuning Sequence.
- d. Display Cycle.

Polarizing Cycle. During this period the sensor or sensors are energized.

The OMNI IV magnetometers utilize the principle of constant energy polarization. Most instruments utilize a fixed polarization time regardless of the battery voltage. It can be demonstrated that with such a method, a 40% deterioration occurs in sensor signal strength rendering the instrument to be more and more inaccurate (scatter phenomena).

The microprocessor within the instrument computes the battery voltage during polarization and at a specific point determines whether or not the polarization time should be increased. This method ensures that the sensor always receives the same amount of energy per unit of time. Although the performance is not linear, a significant improvement results even when the battery is approaching the depletion state.

Signal Analysis Cycle. At the end of the sensor energizing period, the signal decay cycle occurs. The precession signal is monitored and the decay rate, analyzed. This precession signal is compared statistically to an optimized decay curve at 400 points along the curve. Computations are made and the results of the computations displayed as error.

Tuning Sequence. After the signal analysis period, this new tuning value becomes the basis for the next reading. The measurement portion of the reading sequence occupies typically 2 seconds.

Display Cycle. The selected parameter is displayed on the liquid crystal display (LCD) for approximately 8.5 seconds. At that time, the audio circuits provide a sequence of beeps which indicate that the instrument will be shut down after another 8.5 seconds unless the same command or another command is keyed.

Note

During this cycle, valid readings, location and time values are entered into scratch-pad memory as a data block.

To summarize, a typical reading sequence terminates after about 20 seconds. Then the computer turns off the power.

3.6 Audio Circuits

The instrument provides both visual and audio feedback. Every time that a key is pressed, an audio beep will be heard. Parameters are displayed for 8.5 seconds only. The moment that this visual period has elapsed, the audio is activated to give repeated beeps for another 8.5 second period to indicate the end of the data block display period. If the operator fails to press any key during this warning period, the instrument turns off automatically. Every time that a key is pressed, a new 8.5 second period is initiated. This allows the operator to have enough time to evaluate and program data. The display period may be extended by pressing the same key or another key. However, selection of a different key initiates a different display response.

3.7 Statistical Error Alarm

Following each reading, the instrument computes the true statistical error of the total field reading. This value represents the confidence level of the data. A visual alarm may be triggered based on the following criteria:

a. When the statistical error of a particular reading is equal to or larger than 0.2 gamma, the least significant digit of the displayed total field flashes on and off. For example, assume that the statistical error is 0.23 gamma and the displayed total field is: 57936.4. In this case, the 4 flashes on and off. Flashing the least significant digit on and off indicates that the accuracy is impaired and the operator should be warned.

b. When the statistical error of a particular reading is equal to or larger than 2.0 gammas, the two least significant digits of the displayed total field flash on and off. For example, assume that the statistical error is 2.3 gamma and the displayed total field is: 57936.4. In this case, the 6 and 4 flash on and off. Flashing the two least significant digits on and off indicates that the accuracy is badly impaired and an audio warning sounds to alert the operator.

NOTE

If the error is larger than 2.0 gammas, take another reading.

3.8 Automatic Fine Tracking (Tuning) of Total Field

The OMNI IV contains high technology circuits which allow for automatic fine tracking (tuning) over the entire field strength range from 18,000 to 110,000 gammas, under computer control. An optimized tuning algorithm ensures that the system is tuned for optimum performance.

Under normal operating conditions, a $\pm 15\%$ capture tuning range from reading to reading is achieved. The capture tuning range is defined as the difference between the previous and current readings relative to the previous reading. However, if a larger difference is computed, the instrument will warn the operator of this and further measurements are inhibited automatically. The warning is both visual and audible. The TUNE descriptor located on the left hand side of the display flashes on and off and the audio alarm is enabled as well. The displayed total field reading also flashes on and off to inform the operator that the displayed value is invalid. The value displayed, however, is the previous total field value, and should be changed manually to reflect the new tuning field.

3.9 SURVEY GRID

When a total magnetic field survey is conducted, it is necessary to locate and record the ground position where each measurement is made. In the grid method, the operator usually follows a predetermined straight track and takes measurements at fixed intervals along that track. When the arbitrary end of the track is reached, the operator moves a fixed distance to the left or right of this track, then measurements are taken along a parallel track but moving in the opposite direction.

The OMNI IV is designed to operate on a rigidly controlled grid similar to this. See Figure 3-1. The tracks which are followed are designated as lines and the locations along each line where a measurement is taken is designated as a position (station). The fixed distance between each position is defined as the position spacing and the distance between parallel lines is defined as the line spacing.

Preparing the Grid Matrix. When a field measurement is taken, the line number and the position number along that line must be recorded together with the measurement to produce a meaningful record. This recording procedure is simplified by the computer. First the grid coordinates of the starting point are entered. These entries are in the form of a line number and a position number. Then the spacing intervals between lines and between positions are entered by the operator. Therefore, when a reading is taken, the line and position numbers are recorded automatically. At the next position along the line, the computer can update the position number by adding one position spacing increment to the previous position number. This can be accomplished automatically so that the new measurement record is identified by the original line number plus the updated position number.

When the end of a survey line has been reached, the line spacing interval is activated, (either positive or negative as shown in Figure 3-1), to set up the next line number. By the fact that the operator will now be proceeding in the opposite direction, the sign of the position spacing increment must be changed (toggled).

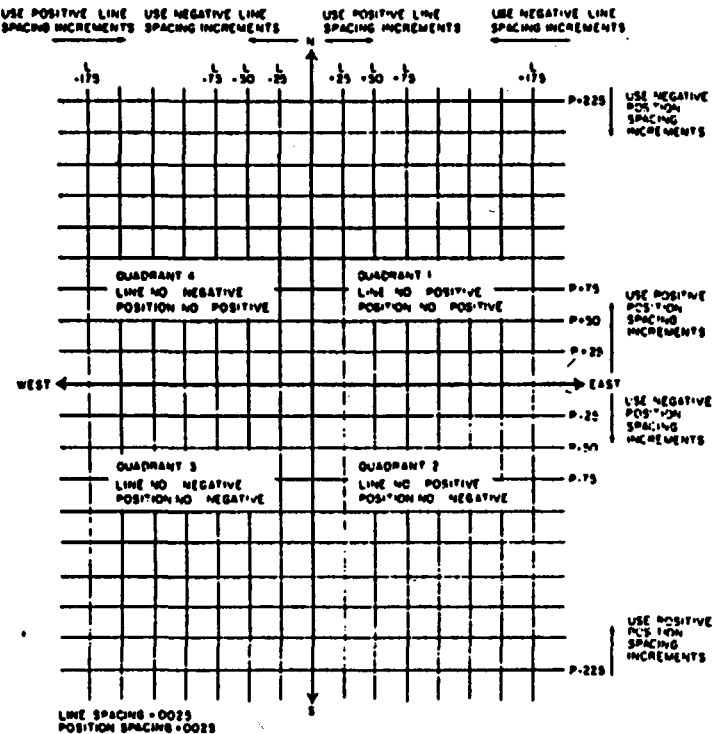


Figure 3-1 Grid Configuration

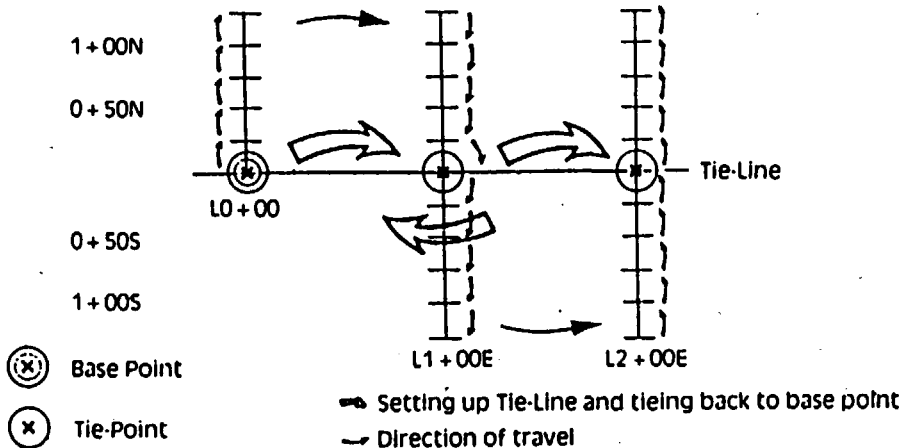


Figure 3-2 Diagram Showing Tie-Line Survey Method

Correction of Survey Data. The self-correcting feature of the OMNI IV is based on using reference measurements obtained by a tie-line or looping method of correcting data. The correction process is accomplished rapidly and accurately using the internal microprocessor, a unique program algorithm and two separate memories.

The procedure is straightforward.

- a. At the beginning of the survey day, a total field reading is taken by the operator at a known and identified base reference point in the survey grid. This is the data and grid reference for either the tie-line or looping method for diurnal correction. The value, which is identified by grid coordinates and the time of the reading, is stored as a data block in a special memory. This special memory is accessed when the MODE selector is set to TIE.
- b. Tie-Line Method. See Figure 3-2. In this method, several coordinates in the survey grid are identified as tie points. Frequently, these tie points are selected along a grid line which passes through the base reference point. However, multiple tie lines or even a random scattering of tie points can be selected.

c. As soon as the base reference recording has been made, a total field reading is taken at each selected tie point in sequence. At the end of the sequence, a second recording is taken at the base reference point to close the loop. This step must be carried out in the shortest time possible to improve the correction accuracy. Each of these data blocks is stored in the tie memory file.

d. Next, the tie point values are corrected diurnally to the base reference point. To achieve proper diurnal correction of each tie point value, the microprocessor computes by linear interpolation, the drift in the earth's magnetic field with respect to the time that each reading was taken. By distributing the drift in the earth's magnetic field and applying it to the times that the tie-point readings were taken, extremely accurate field results are computed for each tie point.

e. Now the remainder of the survey can be conducted by collecting total field values at each coordinate in the survey grid. Each reading is stored as a data block in a separate memory called the field data memory file. This memory is accessed when the MODE selector is set to TFM (for total field magnetometers) or GRAD (for gradiometers). Each time that the operator arrives at a coordinate which has been designated as a tie point, the new reading is flagged in the memory for calculating diurnal drift. The first and last reading of the survey during the day should be on any tie point, in the TFM or GRAD mode; or on the base reference point when the TIE mode is selected.

f. At the end of the survey day, the data stored in the tie-line memory file is merged with the field measurements stored in the field data memory file. The microprocessor then computes the variation in the earth's magnetic field for each grid coordinate and corrects the field data file using the tie-line file. No manual computations are required.

g. Looping Method. See Figure 3-3. This method is similar to the tie-line method. However, instead of selecting numerous tie point references, only the base reference point is used. At the start of the survey, a data block is recorded at the base reference point with the MODE selector set to TIE, and stored in the tie-line memory file.

h. During the survey, total field values are collected at each grid reference. Each reading is stored as a data block in the field data memory file. Then, at intervals within 30 to 60 minutes, the operator returns (loops back) to the base reference point and takes another reading. Each of the total field readings taken at the base reference point is stored as a data block in the tie-line memory file.

j. At the end of the survey day, the base reference data stored in the tie-line memory file is merged with the field measurements stored in the field data memory file.

k. The microprocessor computes by linear interpolation, the drift in the earth's magnetic field with respect to the time that each base reference point reading was taken. Then these corrections are applied to each reading in the field data file.

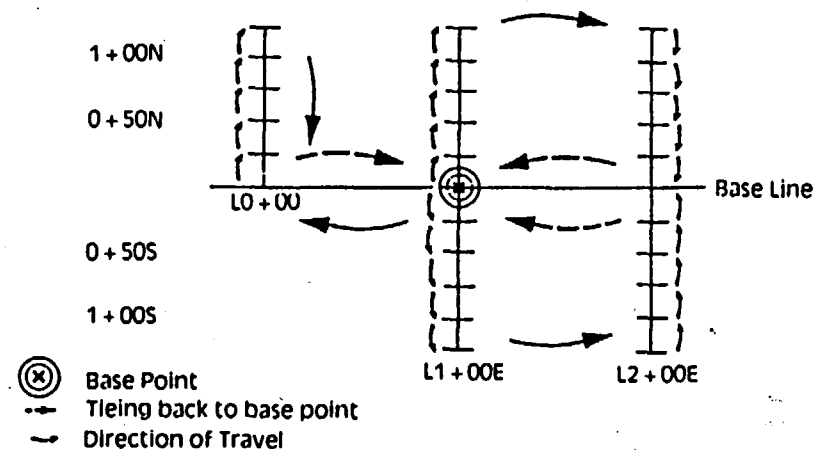


Figure 3-3 Diagram Showing Looping Survey Method

3.10 BASE STATION (AUTO-CYCLING) OPTION

In addition to the self-correcting feature of its data using the tie-line or looping method, the OMNI IV can also include a base station or auto-cycling option. The option enables the instrument to perform as a self-recording base station. This is most useful in locations such as Northern latitudes where more detailed monitoring of the earth's magnetic field is required.

Auto Cycle. The base station OMNI IV is set up and remains stationary at the base reference station. When the MODE selector is set to AUTO, the instrument takes readings at a preprogrammed time interval (adjustable from 5 seconds to 60 minutes in 1 second increments).

Survey Data Correction. In order to correct the total field data recorded in a separate OMNI IV, the total field magnetometer and the base station are interconnected through a cable. This cable is also connected to the external computer or printer. The data from the field unit is then read by the base station and the diurnal variation corrections computed within the base station magnetometer. The base station unit then sends the corrected field data to a printer or a computer.

SECTION 4

OPERATION

4.1 ARRANGEMENT

The electronic console includes:

- a. An operating-mode selector with eight positions.
- b. A programming keypad on the left side, with 12 pressure sensitive keys.
- c. A recording keypad on the right side, with 6 pressure sensitive keys.
- d. An LCD multi-function display with digital readout.

These are all that is necessary for operating and checking the instrument and for making field measurements.

NOTE

(Reference: 1γ (gamma) = 10^{-5} gauss = 1nT (nanoTesla).)

4.2 DESCRIPTION OF SWITCHES AND INDICATORS

The locations of the MODE selector positions, the programming and recording keypad push-buttons, are shown in Figures 4-1, 4-2 and 4-3 respectively. Their functions during normal operation are given in Tables 4-1, 4-2 and 4-3 respectively. The liquid crystal display (LCD) is shown in Figure 4-4. The descriptions are explained in Table 4-4.

4.3 MODE SELECTOR

At the lower edge of the electronic console is an eight position mode selector. The selections permit field measurements, data retrieval, functional testing and battery power conservation.



Figure 4-1 Mode Selector

Description	Function
MODE selector	This eight-position selector allows the instrument to be operated in several different modes (ie, as a field mag, base station, or gradiometer).
OFF	<p>The display heater circuit is disabled when the MODE selector is set to OFF. This prevents unnecessary power drain when the ambient temperature drops below -25°C and the instrument is not scheduled for use for a period of time.</p> <p style="text-align: center;">NOTE</p> <p>The display heater circuit becomes enabled automatically when the temperature is below -25°C and the MODE selector is not set to OFF.</p>
TEST	<p>In this mode, the unit may be tested by pressing the READ key. The instrument conducts a synthetic test to verify proper operation of the electronics and program software. The test result, which is a total test field expressed in gammas, appears on the display, (ie, 56369.7 GAMMA). The program memory is also subjected to a test.</p> <p>In addition, when the ERROR key is pressed, proper operation of the mathematical statistical error routine is verified, (ie, LCD displays .00 GAMMA).</p>
TIE	<p>In this mode, tie-line or looping data is taken and recorded in a separate memory. This becomes the baseline data which is recalled automatically and used by the microprocessor to correct survey readings taken later during the day of survey and in the TFM mode.</p> <p>For TIE, each data block stored in the separate memory contains:</p> <ol style="list-style-type: none"> Time of reading (HH:MM:SS). Total field intensity. Statistical error analysis. Grid coordinates (line number, position number). Decay rate. Signal strength.

Table 4-1 Mode Selector

Description	Function
	NOTE
	Tie-line data can be recalled even if stored on different days.
TFM (Total Field Mode)	<p>This is the selection for normal operation during a survey. In this mode, the instrument may be operated normally to measure and store the total field intensity.</p> <p>For TFM, each data block stored in memory contains:</p> <ol style="list-style-type: none"> Time of reading (HH:MM:SS). Total field intensity. Statistical error analysis. Grid coordinates (line number, position number). Decay rate. Signal strength.
GRAD (Vertical gradient)	<p>This is the gradiometer mode. In this position, two measurements are made:</p> <ol style="list-style-type: none"> The total field intensity is measured using the top sensor. The gradient of the total field is calculated by taking simultaneous readings with both sensors separated by a distance of 0.5 m or 1.0 m. <p>For GRAD, each data block stored in memory contains:</p> <ol style="list-style-type: none"> Time of reading (HH:MM:SS). Total field intensity (top sensor). Gradient measurement. Statistical error analysis. Grid coordinates (line number, position number). Decay rate. Signal strength. <p>In the grad position, the instrument measures, calculates, displays and stores the vertical gradient, expressed in gammas gradient/metre.</p>

Table 4-1 Mode Selector (Cont)

Description	Function
AUTO	<p>This is the base station mode. In this mode, data is taken and accumulated in memory to monitor the earth's magnetic field at the base station location. Readings are taken at a preprogrammed interval. This interval can be varied from 5 seconds to 60 minutes in 1 second increments. Up to 5000 data blocks can be stored in memory. Each data block contains:</p> <ol style="list-style-type: none"> Time of reading (HH:MM:SS). Total field intensity. Difference from the programmed reference field value. Difference from the previous reading (ie, drift). Date of day.
DUMP	<p>This position is applicable to all modes. In this mode, the data is retrieved from the protected memory using various access codes. The access codes format the data output required (eg, field data, corrected data, gradient data, etc). In the data print-out, the grid coordinates can also be represented by compass directions (ie, N, S, E and W).</p>
PROG	<p>This position is applicable to all modes. In this mode, data can be output in an analog profile plot format. Scaling of the profile plot is programmed through the keypads. This selection is ideal for presenting base station data to monitor the activity of the earth's magnetic field. This selection also outputs data in a fixed ASCII format for computer processing.</p>

Table 4-1 Mode Selector (Cont)

4.4 KEYPADS

The console is equipped with two custom-designed keypads: One for programming the instrument; the other for recording and modifying measurement and reference data. The keys are of the touch sensitive type, however, to activate a key some pressure must be applied. An audio tactile feedback system is incorporated so that when a key is pressed, an audio device produces a single short-period beep. The keypads are completely waterproof.

Programming Keypad. This keypad contains 12 keys arranged in a matrix. Keys 1, 2, 3, 5, 6, 9 and CLEAR have three levels; the OFF key has one level; and the remainder have two levels.

a. The first level represents function parameters. These functions are printed in black lettering on a white background (eg, TIME, ERROR, etc).

b. The second level which consists primarily of numerics is printed in red on a white background. The CHANGE key on the recording keypad is the shift key from functions to numerics for the programming keypad.

c. The third level also represents function parameters. These are also shown in black lettering on a white background between the function descriptor and numeric (eg, DATE, DATUM and GRAD).

The OFF key is single level with one function only.

NOTE

The first and third levels form a toggle type push-button. Consecutive operations of the push-button activates the alternate selection.

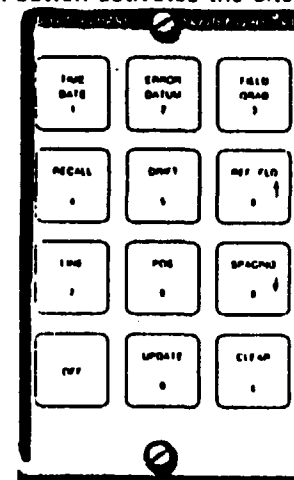


Figure 4-2 Programming Keypad

Key	Function
<div>TIME DATE 1</div>	<p>When this key is pressed once, the actual time of day (ie, 24 hour real-time clock) will be displayed in hours, minutes and seconds (HH:MM:SS) (eg, 13:24:42). The time is not updated automatically on the display. The displayed time relates to the time of a measurement.</p> <p>When the key is pressed a second time, the date will be displayed in year, month, day (YY:MM:DD) (eg, 85:03:31). (The clock does not have leap year compensation. The year must be updated manually on Jan 1st.) When changing the date, the time must also be re-entered in order to enter the new date in the real-time clock.</p> <p>When any displayed parameter requires changing and the instrument is in the change mode (ie, when the CHANGE key is pressed), the TIME key becomes a numeric key with a value of 1.</p>
<div>ERROR DATUM 2</div>	<p>When the key is pressed once, the statistical error will be displayed in gammas, with a resolution of 0.01 γ.</p> <p style="text-align: center;">NOTE</p> <p>The statistical error can only be displayed after the total field value has been displayed. Refer to FIELD key operation. To display the statistical error, the ERROR key may be pressed at any time to obtain a first time display or for display recall purposes.</p> <p>When the key is pressed a second time, a datum value can be entered. The datum is then subtracted from the raw or corrected total field data when it is being dumped.</p> <p>When any displayed parameter requires changing and the instrument is in the change mode (ie, when the CHANGE key is pressed), the ERROR key becomes a numeric key with a value of 2.</p>

Table 4-2 Programming Keypad

Key	Function
<div>FIELD GRAD 3</div>	<p>The FIELD and GRAD selections may be considered as a toggle switch. When this key is pressed once, the total magnetic field reading will be displayed, expressed in gammas.</p> <p style="text-align: center;">NOTE</p> <p>This key is used primarily for total field recall, because, in normal operation, the total field value will be displayed first automatically.</p> <p>This key may also be used for displaying the total field value when it requires changing whenever the instrument needs to be tuned.</p> <p>When the GRAD mode is selected and this key is pressed, the display first indicates total magnetic field reading taken by the upper sensor. When the key is pressed again, it toggles to the GRAD selection and displays the vertical gradient automatically in gammas gradient/metre. The gradient display cannot be changed.</p> <p>When any displayed parameter requires changing and the instrument is in the change mode (ie, when the CHANGE key is pressed), the FIELD key becomes a numeric key with a value of 3.</p>
<div>RECALL 4</div>	<p>This selection accesses data in memory to recall the total field readings entered previously. With RECALL selected keys 6 and 9 can be used to scroll the memory in order to display previous records. Key 9 decrements the record location; key 6 increments it. In recall, any stored parameter (ie, date, time, line, position, error, total field, or gradient) can be displayed by pressing the appropriate function key.</p> <p style="text-align: center;">NOTE</p> <p>During recall, the decay and sensor descriptors will all be flashing to distinguish between parameters displayed in normal operation and those displayed in recall.</p>

Table 4-2 Programming Keypad (Cont)

Key	Function
<div>DRIFT</div> <div>5</div>	<p>When any displayed parameter requires changing and the instrument is in the change mode (ie, when the CHANGE key is pressed), the RECALL key becomes a numeric key with a value of 4.</p> <p>When this key is pressed once, the change in reading between the two most recent readings is displayed. When the key is pressed again, it indicates the field variation between the two most recent tie points.</p> <p>When any displayed parameter requires changing and the instrument is in the change mode (ie, when the CHANGE key is pressed), the DRIFT key becomes a numeric key with a value of 5.</p>
<div>REF.FLD</div> <div>6</div> <div>↑</div>	<p>When this key is pressed in the base station or tie-line mode, a programmable magnetic field value can be entered. This value is then subtracted from the raw base station data to obtain the drift correction which is to be applied to the field data. The reference field is also used for the drift correction if the unit is used as a tie-line magnetometer.</p> <p>When RECALL has been selected, this key (1) increments the memory location. When the key is held, it scrolls the memory in the fast forward direction.</p>
<div>LINE</div> <div>7</div>	<p>When this key is pressed once, the current line number (longitude) will be displayed. This is a four digit number and can be either positive or negative. For example, a line to the west of the quadrant reference might display L-0525.</p> <p>When any displayed parameter requires changing and the instrument is in the change mode (ie, when the CHANGE key is pressed), the LINE key becomes a numeric key with a value of 7.</p>
<div>POS</div> <div>8</div>	<p>When this key is pressed once, the current position or station number (latitude) will be displayed. This is a four digit number and can be either positive or negative. For example, a position to the south of the quadrant reference might display P-0085.</p>

Table 4-2 Programming Keypad (Cont)

Key	Function
<div>SPACING</div> <div>9</div> <div>↓</div>	<p>When any displayed parameter requires changing and the instrument is in the change mode (ie, when the CHANGE key is pressed), the POS key becomes a numeric key with a value of 8.</p> <p>In field survey applications, (ie, TIE, TFM or GRAD), this key initiates a display of the spacing between lines or positions.</p> <p>a. To obtain line spacing:</p> <p>(i) The LINE key is pressed first. This causes the current line number to be displayed.</p> <p>(ii) Next, the SPACING key is pressed to display the spacing increment between adjacent lines which has been set into the instrument memory. An example of a spacing display is INTV L-0025.</p> <p>NOTE</p> <p>When line spacing is being displayed, the descriptor INTV (interval) appears on the left of the display. This enables the operator to differentiate between a line number display and a line spacing display.</p> <p>b. To obtain the position (station) spacing:</p> <p>(i) The POS key is pressed first. This causes the current position number to be displayed.</p> <p>(ii) Next, the SPACING key is pressed to display the spacing increment between adjacent positions which has been set into the instrument memory. An example of a spacing display is INTV P-0025. This means that for each sequential field measurement, the grid position reading will change by -0025. Station spacing of 12.5 can be entered by keying in 0012. The display will show 00:12 with both the decimal point and colon flashing to indicate that the .5 is not being displayed.</p>

Table 4-2 Programming Keypad (Cont)

Key	Function
	<p>NOTE</p> <p>When position spacing is being displayed, the descriptor INTV (interval) appears on the left. This enables the operator to differentiate between a position number display and a position spacing display.</p> <p>In base station applications, (ie. AUTO mode), after first pressing the TIME key, the SPACING key initiates display of the programmed sampling interval (ie. set between 5 seconds and 60 minutes). Time spacing is also used for the field magnetometer if synchronization is required between the base station and field units. If it is not required, this feature is disabled by entering 0000 for the time spacing in the field unit.</p> <p>When RECALL has been selected, this key (I) decrements the memory location. When the key is held, it scrolls the memory in the fast backwards direction.</p> <p>When any displayed parameter requires changing and the instrument is in the change mode (ie, when the CHANGE key is pressed), the SPACING key becomes a numeric key with a value of 9.</p>
<div style="border: 1px solid black; border-radius: 10px; padding: 5px; text-align: center;"> UPDATE 0 </div>	<p>The function of this key is to update the displayed position number in the survey grid. When the UPDATE key is pressed and no parameter related to grid coordinates is displayed, as a default, the position number will be incremented or decremented automatically defined by the position spacing and sign. To update the previous position number, the operator may also press the POS key first. The display will indicate the last stored position number. When the UPDATE key is pressed, the displayed position number is updated by the programmed position spacing increment.</p> <p>Consider the example in which the current position number in the grid is P-0350; and the position spacing increment is INTV P-0015.</p>

Table 4-2 Programming Keypad (Cont)

Key	Function
	<p>NOTE</p> <p>In terms of the grid reference, this implies that the operator is south of the zero position reference and is proceeding southwards along a grid line. If the POS key is pressed, P-0350 will be displayed. Then, when the UPDATE key is pressed, the spacing increment will be added to this: (ie, $-0350 + (-0015) = -0365$). The display then changes to P-0365. This is the position number of the ordinate which is adjacent to the updated ordinate along the track.</p> <p>Consider another example with P 0575 (ie, north of the zero reference line) and INTV P-00:12 (ie, proceeding southwards). The updated position number becomes $+0575 + (-0012.5) = P 0562.5$. This is displayed as P 05:62.</p> <p>The procedure for updating the line number by adding the line spacing is the same provided that the current line number is displayed first, (ie, press LINE prior to pressing the UPDATE key). It is evident that all four quadrants on the survey grid can be described accurately provided that the origin of the grid is in the centre.</p>
	<p>NOTE</p> <p>If the display is not indicating a position number or the position spacing when the UPDATE key is pressed once, the display will indicate automatically the updated position number on the grid as a default condition.</p> <p>When any displayed parameter requires changing and the instrument is in the change mode (ie, when the CHANGE key is pressed), the UPDATE key becomes a numeric key with a value of 0.</p>

Table 4-2 Programming Keypad (Cont)

Key	Function
<div>CLEAR</div> <div>±</div>	<p>In any of the manual measurement modes, (ie, TIE, TFM or GRAD), if the RECALL key is pressed to display the most recent reading, then the CLEAR key is pressed, that reading is deleted from the memory. No other reading can be deleted.</p> <p>In the DATA DUMP mode, if the CLEAR key is pressed it clears the tie-line memory of all tie-line data if HP8888 is entered.</p> <p>When the instrument is in the change mode (ie, when the CHANGE key is pressed), the CLEAR key becomes a mathematical key with a ± toggle application to change the sign.</p> <p style="text-align: center;">NOTE</p> <p>Only the negative sign is displayed. The positive sign is replaced by a space or blank.</p>
<div>OFF</div>	<p>When this key is pressed, it commands the instrument to turn off power. The power-down sequence is under computer control to ensure an organized shut-down.</p> <p style="text-align: center;">NOTE</p> <p>The OMNI IV shuts down automatically after approximately 20 seconds if no keys are pressed. The OFF may be used to override the normal 20 second display time-out period to conserve battery power any time.</p>

Table 4-2 Programming Keypad (Cont)

Recording Keypad. This keypad contains 6 keys in a matrix. The READ and AUTO-RECORD keys occupy the space of two square keypads. The READ key has black lettering on a bright red background. These keys are single level. The SPOT, MULTI and AUTO RECORD keys enable the operator to take a total field reading and record the data in a choice of three different ways depending upon the survey grid configuration established:

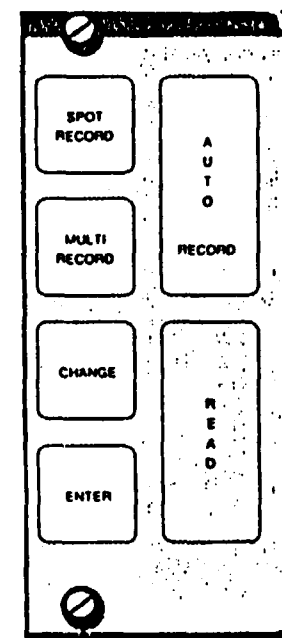


Figure 4-3 Recording Keypad

Key	Function
	<p align="center">NOTE</p> <p>The SPOT RECORD, MULTI RECORD and AUTO RECORD keys are single level keys with a built-in safety mode. Each key has to be pressed twice before an action can be initiated. Following a data dump sequence, and when the operator attempts to store the very first reading again using the SPOT RECORD, MULTI RECORD or AUTO RECORD keys, the instrument will refuse to store the data block unless the software code plus an operator code number has been entered.</p>
READ	<p>When this key is pressed, it commands the instrument to turn on its power. First, the computer initializes itself, then the setting of the MODE selector is read. The computer program then proceeds with routines on the basis of the MODE selected. Then the OMNI IV takes a reading of the magnetic field and displays it on the LCD. This reading can then be stored using one of the three RECORD methods.</p>
SPOT RECORD	<p>In this record, the information is stored together with a record pointer number. There is no indication of the grid coordinates (ie, line and position number). Following a data dump, the first spot record becomes 0001. This is incremented with each new reading. When SPOT RECORD is pressed once, the next record number is displayed. A typical display could be RP0052. (The prefix designator RP means record pointer.) This RP number becomes assigned automatically to the data to be stored in memory.</p> <p>When the number is displayed, it allows the operator to record this number, on a map or chart for example, for cross-reference later when required. The new data will not be stored in memory yet. To store the data under the displayed record pointer, the SPOT RECORD key must be pressed a second time. In response, the instrument stores the data, then it switches itself off immediately.</p>

Table 4-3 Recording Keypad

Key	Function
MULTI RECORD	<p>This key is used to store many readings at one station without updating the station coordinates. The multiple entries are similar to the SPOT RECORD entry except that the coordinates are repeated. At the appropriate grid coordinates, the READ key must be pressed to obtain the total field reading. This reading is displayed on the LCD. When the MULTI RECORD key is pressed once, the position number will be displayed and an audio beep is repeated until a follow-on key operation occurs, or the wait period times out. To store the data under the current grid coordinates, the MULTI RECORD key must be pressed a second time. In response, the instrument stores the data, then it switches itself off immediately. Repeat readings can be taken and stored by pressing READ, then MULTI RECORD twice. However, the data printout will show these readings keyed to the same grid coordinates (ie, the coordinates do not increment in this multi record mode). With the MODE selector set to TIE, this key functions in the same way, except that the default parameter displayed is the LINE instead of the POSITION.</p>
AUTO RECORD	<p>This key is used in two modes: Tie line measurements; and field survey measurements.</p> <ol style="list-style-type: none"> When TIE is selected and a reading is taken, the line number is incremented automatically by the programmed position spacing increment. When TFM or GRAD is selected and a reading is taken, the position number is incremented automatically by the programmed line spacing increment. <p>At the appropriate grid coordinates, the READ key must be pressed to obtain the total field reading. This reading is displayed on the LCD. When the AUTO RECORD key is pressed once, the line number will be displayed (in the TIE mode) and the position number will be displayed in the TFM or GRAD modes. At the same time an audio beep is repeated until a follow-on key operation occurs, or the wait period times out. To store the data under the current grid coordinates, the AUTO RECORD key must be pressed a second time. In response, the instrument stores the data, the position number (TFM or GRAD) or the line number (TIE) increments by the spacing increment, then it switches itself off immediately.</p>

Table 4-3 Recording Keypad (Cont)

Key	Function
	<p>NOTE</p> <p>If interaction is required to change the displayed coordinate, this can occur before the key is pressed for the second time because the data will not have been stored in memory yet.</p>
CHANGE	<p>When the CHANGE key is pressed, two functions are initiated:</p> <p>(i) The keypad function keys are changed to the numeric value mode.</p> <p>(ii) The values of the following parameters can be selected, displayed and changed individually: Time; date; total field, (for the purpose of tuning only); line number and line spacing; position number and position spacing; operator number; and confidential number.</p>
ENTER	<p>This key enters the desired numeric data into memory and returns the selected function key to the function (black) level. This key can be pressed at any time, even while some digits are still flashing. When the audio warning is sounding and all digits are flashing, this key is disabled until after the CHANGE key is pressed first to turn off the audio warning.</p>

Table 4-3 Recording Keypad (Cont)

4.5 LIQUID CRYSTAL DISPLAY (LCD)

The display is a custom-designed, ruggedized, high-quality, liquid crystal display. It utilizes a fluid which allows the display to be operated in temperatures as low as -35°C without any heating. However, at -30°C , there is already a significant segment turnover slow-down. A safety margin is provided by incorporating a heater system which becomes enabled at about -25°C . During prolonged low temperature operation, the heater circuitry remains enabled even when the power to the instrument is switched off, provided that the MODE selector is not set to OFF.

NOTE

When the ambient temperature is lower than -25°C and the magnetometer is not in use for a while, it is recommended that the MODE selector be set to the OFF position. This action prevents continuous battery power drain. The display comprises ten individual descriptors; three bar and wedge graphic indicators; and the primary numeric readout. The numeric readout consists of six digits; two colons which separate the digits into three pairs; and two individual decimal points which indicate tenths or hundredths. (Examples of displays are given throughout Tables 4-1 and 4-2.) Refer to Figure 4-4 and Table 4-4.

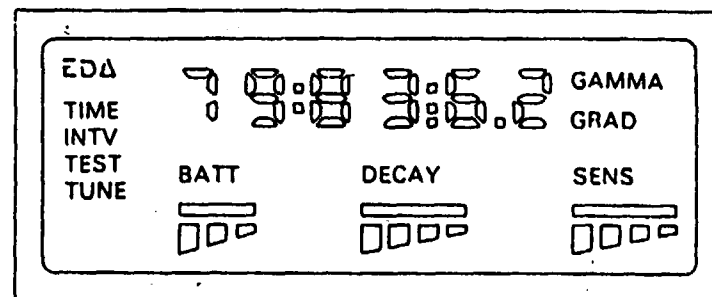


Figure 4-4 Liquid Crystal Display

Description	Function
EDA logo	<p>The letters EDA are displayed whenever the instrument is on. The logo will commence to flash on and off during data dumping when the internal lithium battery system which is for emergency battery back-up, is depleted. When the logo starts to flash, the lithium battery must be replaced.</p> <p style="text-align: center;">NOTE</p> <p>EDA recommends that the internal lithium battery be replaced by EDA personnel. If this is not possible, consult EDA first, prior to changing the battery, to obtain instructions. The life of the lithium battery is estimated to be five years under normal conditions.</p>
TIME	The word TIME will be displayed when the TIME key is pressed once. This is accompanied by the six-digit local time display. When the TIME key is pressed a second time, the word TIME disappears and the date is displayed.
INTV	The descriptor INTV (interval or spacing) will be displayed when the time interval, line or position spacing is displayed.
TEST	The word TEST will be displayed when the MODE selector is set to the TEST position and the READ key is pressed.
TUNE	The word TUNE will be displayed in a flashing manner automatically when the instrument detects that the system requires tuning. Tuning is achieved by pressing the CHANGE key and entering a new value of total field which is close to the actual ambient total field value.
GAMMA	The word GAMMA will be displayed when the actual total field measurement or the test field reading is displayed. The word also appears when the ERROR key is pressed.
GRAD	The word GRAD will be displayed when the MODE selector is set to the GRAD position, and the gradient is displayed by pressing the FIELD/GRAD key. The word GRAD will also be displayed if the error of the gradient (lower) sensor is displayed.

Table 4-4 Liquid Crystal Display

Description	Function
BATT	<p>The word BATT will be displayed during normal operation. It indicates that adequate power remains in the battery system. When BATT commences to flash on and off, the operator is warned that the battery is approaching the discharged condition rapidly. When the audio alarm is activated, the battery must be changed or charged. No further readings are possible.</p> <p>The battery monitor comprises the BATT descriptor, a rectangle and three wedge-shaped bars. With a fully charged battery system, all three bars are displayed. Gradually, after many readings, the residual battery power decreases. This is indicated when the left hand bar disappears. With continued battery discharge, the second bar disappears, followed eventually by the third bar. When this condition occurs, the battery is almost depleted. Therefore, it is recommended that the battery system be recharged or replaced when the BATT descriptor commences to flash on and off. The instrument inhibits any further readings when the BATT descriptor flashes and the audio alarm is actuated.</p>
DECAY	<p>The word DECAY will be displayed during normal operation. When the word commences to flash on and off, it indicates that the decay rate of the precession signal is too fast due to large local gradients.</p> <p style="text-align: center;">NOTE</p> <p>A possible way to reduce the effect of the large local gradient is to raise the sensor.</p> <p>The precession signal decay rate monitor comprises the DECAY descriptor, a rectangle and four wedge-shaped bars. Every time that a reading is taken, the computer calculates the actual decay rate. This is an important parameter because it reveals the degree of gradient tolerance. When the decay rate is too fast due to large local gradients, the accuracy of the measurement is impaired to some degree. The statistical error value reveals this immediately. All four</p>

Table 4-4 Liquid Crystal Display (Cont)

Description	Function
SENS	<p>wedge shaped bars will be displayed when a normal decay rate is computed. When the operator gradually approaches a large local gradient, the computed decay rate increases, and the wedge-shaped bars may gradually disappear in sequence, from the left until the decay rate is too fast for proper signal processing. When this occurs, the DECAY descriptor commences to flash on and off and completion of the reading is inhibited. The reading is aborted and the TUNE descriptor flashes on and off.</p> <p>The word SENS will be displayed during normal operation. When the word commences to flash on and off, it indicates that the starting amplitude of the precession signal is below the minimum acceptable threshold. This condition may occur when:</p> <ol style="list-style-type: none"> The sensor is damaged (fluid in sensor is gone). A very large local gradient is encountered. Sensor is not properly aligned, especially in areas where the total field vector inclination angle is less than 45°. <p>The sensor signal monitor comprises the SENS descriptors, a rectangle and four wedge-shaped bars. Every time that a reading is taken, the computer calculates the actual starting amplitude of the precession signal derived from the sensor. This monitor may be used to ensure that the sensor is aligned properly with magnetic North.</p> <p>When the signal amplitude is normal and above a pre-defined threshold, all four wedge-shaped bars are displayed. If the calculated amplitude decreases, the bars disappear in sequence from the left until the amplitude does not meet the minimum threshold level. When this occurs, the SENS descriptor commences to flash on and off.</p>

Table 4-4 Liquid Crystal Display (Cont)

4.6 PREPARATION FOR USE

Unpacking. Remove all components from the shipping container(s) and check the contents against the Packing Slip/Release Note. Report any shortages or damage immediately to EDA Instruments Inc.

Battery Packs. There are two battery configurations with various battery types. Refer to Table 2-1. One battery pack attaches directly to the underside of the instrument so that it is an integral part when the instrument is chest mounted. Another battery arrangement has the batteries in a wide web belt which is attached around the waist. The connecting cable for the belt pack terminates in a base plate which is similar in appearance to the integral battery pack.

- Secure the battery pack (or the belt connector-base plate) to the base of the console. Align the contact springs in the pack with the conductive discs in the base of the console. (Note: The plate can be installed with the attaching screws at either end.)
- Tighten the four captive screws in the plate finger tight.
- Buckle the pack around the waist and adjust by tightening the strap.

Console. The instrument console is carried on the chest and secured by a dual shoulder harness.

- Attach the harness to the instrument then adjust the straps so that the console is at a suitable viewing and operating height.

Sensor. There are two connectors on the base of the instrument: The one adjacent to the left hand when the instrument is chest mounted is for the sensor cable; the one adjacent to the right hand is for the computer or printer interface cable used to dump data.

- Attach the cable to the left hand connector. The connector is keyed (ie, centre pin blocked) so that the sensor will only mate with one connector.
- Mount the sensor on the top section of the pole or staff. The end terminates in a tapered, threaded adapter.
- Connect the other pole sections in series with the white plastic end tip at the base of the pole.

Operating Stance. To take field measurements, hold the sensor pole vertically at arm's length away from the console. Then operate the appropriate keys.

4.7 OPERATING PRECAUTIONS AND GUIDELINES

☐ **Metallic Objects.** Prior to using the OMNI IV, remove all magnetic material from the body. (To check for the presence of such materials, take two sample readings without moving the sensor. Press the READ key, then press the FIELD key. Take one reading while standing near the sensor, and the other reading at some distance from the sensor. If there is no change in the readings, there is no magnetic material present. Do not store the readings.)

☐ **Survey Log.** Maintain a notebook record during the survey. Annotate all errors and deviations from the usual numbering sequence, SPOT records, etc. Record the locations of roads, fences, pipes, houses. Without clear records, numbering errors are nearly impossible to recover.

☐ **Survey Grid.** Maintain a constant check on the surveyed line and station locations. It is very easy to update position numbers inadvertently, especially when taking multiple readings at a station. It is much more difficult to correct these errors once they are stored in memory. Do not let errors propagate.

☐ **Ensure that four-digit line numbers and position numbers describe all station coordinates adequately including fill-ins.**

☐ **Spot Records.** Use spot records to delimit erroneous records. For example, if several station recordings have been taken erroneously, take a spot record before resuming the proper sequence. Annotate this reading in the log notebook together with a description of the problem.

☐ **Time Clock.** Synchronize the instrument clocks daily. Drift is ± 1 sec per 24 hours. Make sure that the date is the same on both instruments.

☐ **Base Stations.** When a base station instrument is being used, revisit the base station once a day to verify that it is cycling. Wait for the period to time out to verify that it is functioning. If it has stopped, it should restart by pressing the READ key. If the fault repeats, re-initialize the unit.

☐ **When an operator returns to the base station at the end of the day, if the base station instrument has shut down, be sure to take some readings as follows:**

a. Wait for cycling interval to time out before deciding that the base station unit has shut down.

b. Set the MODE selector to TFM.

c. Take several readings and store them.

☐ **Initialization.** Read and understand the initialization procedure for field operations.

CAUTION

Never initialize the OMNI IV when it contains valid stored data. This will clear the memory.

☐ **Performance Monitoring.** Observe the DECAY and SENS wedged-shaped bars on the LCD continually, because these give a quick indication of a malfunction. It is possible to observe factors which are degraded severely in areas of high magnetic gradient; so much so, that it may be impossible to store the reading.

☐ **Battery Duration.** A fully charged belt pack will provide power all day (2.5 amp-hours) if total field data is collected, or 6 hours if gradient data is collected. Twelve disposable D cells will provide 5 amp-hours of power which is good for up to 3 days of survey based on 1500 to 2000 readings per day. If a battery does not have sufficient power to achieve constant sensor polarization, the OMNI IV shuts down. If the OMNI IV completes a reading, then that reading can be considered good regardless of the indication of the BATT wedge-shaped bars.

4.8 OPERATING PROCEDURES

CAUTION

At all times, ensure that no device other than the sensor cable is connected to the sensor connector on the OMNI IV. The data dump connector is not capped, therefore, ensure that dirt or contaminant does not damage it.

Similar to most microprocessor-oriented systems, operating procedures are primarily at the discretion of the operator. Keypad control provides the flexibility feature which is only limited by operator ingenuity.

An example grid survey is summarized in Appendix A. In the following paragraphs, the following operating topics are discussed:

General Procedures (para 4.8.1).

Starting the Unit (para 4.8.2).

Changing a Parameter (para 4.8.3).

TEST Mode (para 4.8.4).

Total Field Test (para 4.8.5).

Error Calculation Test (para 4.8.6).

Software Diagnostics (para 4.8.7).

Making Initial Total Field Measurements (para 4.8.8).
 Statistical Error Evaluation (para 4.8.9).
PRE-SURVEY PROCEDURES (para 4.9).
 Identifying the Instrument (para 4.10).
 Erasing the Previous Day's Survey Data (para 4.11).
 Initializing the OMNI IV (para 4.12).
 Tie-Line Memory (para 4.12.1).
 Field Memory (para 4.12.2).
 Initializing the Field Memory (para 4.12.3).
 Initializing the Tie-Line Memory (para 4.12.4).
 Starting the Base Station Magnetometer (para 4.12.5).
 Starting the Gradiometer (para 4.12.6).
Pre-Survey Programming (para 4.13).
 Real-Time Clock (para 4.13.1).
 Reading Synchronization (para 4.13.2).
SYSTEM TUNING (Prior to Survey) (para 4.14).
 Retrieval of Last Previously Stored Total Field Value (para 4.14.1).
 Tuning the OMNI IV (para 4.14.2).
 Incomprehensible Displays (para 4.14.3).
SYSTEM TUNING (During a Survey) (para 4.15).
 Large Gradients (para 4.15.1).
PREPARING THE SURVEY GRID PROFILE (para 4.16).
 Survey Track (para 4.16.1).
SETTING UP A TIE LINE (para 4.17).
 Extending the Tie Point Grid (para 4.17.1).
 Displaying the Tie Point Drift (para 4.17.2).
SETTING UP A LOOP SURVEY (para 4.18).
FIELD MEASUREMENTS (para 4.19).
 Erasing Field Data Records (para 4.20).
 Choosing Recording Format (para 4.21).
 Skipping Stations (para 4.22).
 Intermediate Points (para 4.23).

4.8.1 General Procedures. The following procedures apply to keying operations which can be carried out at any time, and do not necessarily relate to specific survey procedures. These procedures also apply to the OMNI IV regardless of the software level (ie, field mag, base station and gradiometer).

4.8.2 Starting the Unit. When a charged battery is connected, the OMNI IV is ready to operate.

- a. Set the MODE selector to the required position.
- b. Press the READ key. This turns on the power to the system.

4.8.3 Changing a Parameter. Whenever a parameter key is pressed, the stored value of that parameter will appear on the display. To change a parameter value (such as TIME, DATE, LINE, POS, LINE SPACING, POS SPACING, or TUNING):

- a. Press the parameter key (eg, TIME).
- b. Then press the CHANGE key. This causes the displayed value to flash on and off, indicating that the value may now be changed.
- c. If it is applicable to the value, press the +/- key.
- d. Press the required numeric keys in sequence from left to right to make up the new number.

NOTE

As each digit is entered, the numeric stops flashing.

e. When the new parameter value is being displayed, verify its accuracy, then press the ENTER key. The new value then enters the OMNI IV memory.

f. Press the parameter key again and verify on the display that the correct value has been entered and stored.

NOTE

If a keying error occurs when entering the numerics, press the CHANGE key again.

4.8.4 TEST Mode. The OMNI IV circuitry and software includes a built-in test capability which confirms the validity of field measurements, data acquisition and record processing. The following tests are available:

- a. Total field test.
- b. Error calculation test.
- c. Software diagnostics.

NOTE

1. Repeated test readings performed under c. allow for hard-copy verification that the OMNI IV is operating properly.
2. About 85% of the OMNI IV electronics is tested in the TEST mode.
3. These tests may be repeated several times with the OMNI IV in any position to verify repeatability. Once repeatability has been confirmed, the OMNI IV is now ready to be used for a survey provided that certain survey grid parameters (or base station recording interval) are pre-programmed into the memory. At random intervals during a survey, the total field test and error calculation test may be repeated to verify that the OMNI IV is operating properly during the actual survey.

4.8.5 Total Field Test. Proceed as follows:

- a. Set the MODE selector to TEST, then press the READ key. On the display, the EDA logo and GAMMA descriptor will appear. Also the word TEST will flash on and off.
- b. The OMNI IV will take a synthetic reading and complete a total field test. The display will indicate a six-digit numeric value which represents the test total field. The displayed field value is 56369.7 GAMMA.

NOTE

As a result of this test, there is an 80% probability that the OMNI IV will operate satisfactorily.

4.8.6 Error Calculation Test. This is a total field (or gradient) error calculation test. Proceed as follows:

- a. Leave the MODE selector on TEST.
- b. Press the ERROR key once. The display must indicate a value of .00 GAMMA.

c. For gradiometers, (ie, software 5), press the GRAD key once. The display must indicate a value of .0 GAMMA GRAD.

d. Press the ERROR key once. The display must indicate a value of .00 GAMMA GRAD.

4.8.7 Software Diagnostics. A feature is included whereby the OMNI IV executes a complete verification program of the stored data by computing and storing the check sum pertaining to a data block. This feature enhances stored data validity. The check sum verification program checks if there are any sudden problems in the stored program memory.

The program memory diagnostics program is initiated in the DUMP mode. Proceed as follows for the DUMP mode:

- a. Set the MODE selector to DUMP.
- b. Press the READ key. Observe the display. The display indicates: NNNNNN (ie, a six digit number. Refer to para 4.10 for interpretation).

Note

For this test, it is necessary to know the unit serial number.

- c. If the value during the display is correct, the program memory is satisfactory.
- d. If the value during the display is alphanumeric or incomprehensible, record this because there is a problem in the program memory.

4.8.8 Making Initial Total Field Measurements. Proceed as follows:

- a. Refer to the cardinal point decal on the end of the sensor to establish the North direction of the sensor.
- b. When a remote sensor is used, ensure that the sensor is connected to the OMNI IV and keep the sensor as far away as possible from the OMNI IV.

CAUTION

Ensure that the sensor pole is not in the vicinity of magnetic objects or power lines.

- c. Rotate the sensor to align it with magnetic North.

NOTE

The OMNI IV is very sensitive and capable of measuring variations of 0.05 gamma.

- d. Set the MODE selector to TFM.

e. Press the READ key once. The OMNI IV will power-up automatically, then it commences to energize the sensors for a specific polarization period. During this period, the display will indicate a six digit total field value with the two most significant digits flashing on and off. For example, the display may indicate 58315.6 GAMMA and digits 5 and 8 flash on and off. The six-digit value (58315.6 γ) is the last previously-stored value. The flashing 58 indicates that the OMNI IV is tuned to $58 \times 1000 \gamma$. This is also applicable when the MODE selector is set to the GRAD position.

f. After about 2 seconds, the display will be updated automatically. During the brief display update, an audio beep will be activated. The new numeric value on the display will be the actual total field reading obtained, eg, 58186.2 GAMMA, when in the TFM mode, or eg, 2.7 GAMMA GRAD when in the GRAD mode.

g. The actual total field or gradient value will remain on the display for about 8.5 seconds. After this, the audio beep is enabled for a period of 8 seconds to warn the operator that within another 8.5 seconds, the OMNI IV will shut itself off automatically.

h. During the entire display period, other parameter keys may be pressed to display the value for that parameter. Every time another parameter key is pressed, a new 17 second display period will be activated. The display periods for each parameter should be minimized to conserve battery energy.

4.8.9 Statistical Error Evaluation. When in the GRAD mode and after sensor polarization, the display is updated to indicate the value of the gradient. In relatively low noise and gradient free areas, errors of .03 to .07 gamma are typical for the total field value.

a. To display the error value of the gradient (lower) sensor, press the ERROR key. Both the GAMMA and GRAD descriptors will be displayed.

b. To display the error of the total field (upper) sensor, it is required to display the total field first by pressing the FIELD key.

c. Once the total field is displayed, the error of the total field can be displayed by pressing the ERROR key. Only the GAMMA descriptor will be displayed when displaying the total field error.

NOTE

In the gradient mode, only the error of the gradient (lower) sensor is stored.

4.9 PRE-SURVEY PROCEDURES

At the start of a survey day, prior to commencing operations, it is necessary for the operator to perform a start-up procedure. This is to ensure that stored data is not destroyed; that accurate measurements can be taken; and new data will be stored properly for retrieval later. Carry out the following procedures:

- a. Identify the OMNI IV correctly by identifying the software that is resident in the instrument (ie, what modes of operation can be initiated and performed).
- b. Capture Stored Data. Ensure that accumulated data stored from the previous day is dumped prior to initializing the OMNI IV.
- c. Initialize the OMNI IV memories for the current day's survey activity.

CAUTION

1. Initialization of the OMNI IV clears any stored data from the memories, except tie-line data.
2. All OMNI IV magnetometers have a software flag at 12:00 midnight. Data cannot be collected and stored past 12:00 midnight without first dumping (or clearing) the stored data which was collected prior to midnight. This ensures that data which has been collected on a specific day can only be corrected for diurnal variations which have been measured (or computed) during the same specific day.

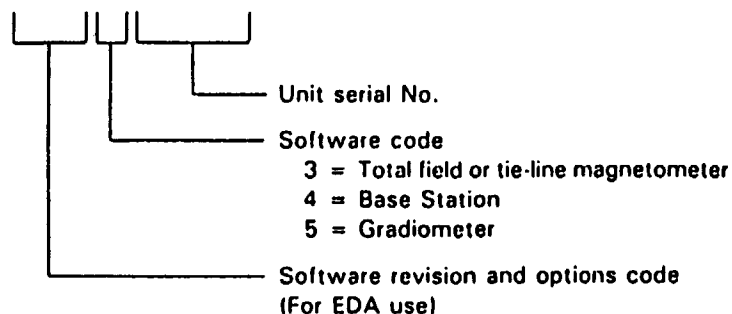
4.10 Identifying the Instrument

If there is more than one instrument being used in a survey, (eg, base station and total field magnetometers), it will be necessary to identify the capabilities of the unit by determining the software that is resident in the unit.

- a. Turn the MODE selector to DUMP.

- b. Press READ key. A six-digit number will be displayed on the LCD.

eg. 063108



4.11 Erasing the Previous Day's Survey Data

At the beginning of each day's survey, it is necessary to initialize the OMNI IV by entering an operator's code 'OPNNNN' where N = a numeric. If data is still in the memory from the previous day, it will be erased when the operator's code is entered.

4.12 Initializing the OMNI IV

The OMNI IV has two memories for storing data: The tie-line memory and the field memory.

4.12.1 Tie-Line Memory. This memory stores data for up to 100 tie points and the base reference. This data in memory is not destroyed until the operator initializes the tie-line memory. The data is retained for tie-line survey use until it is initialized regardless of the number of days the survey might take. This memory is entered in the TIE mode.

4.12.2 Field Memory. This second memory stores the field data. It is initialized when the operator's code number has been entered and the first total field reading has been stored. This memory is entered in the TFM, GRAD, or AUTO modes.

CAUTION

After this point, the prior day's field data is lost and cannot be dumped.

4.12.3 Initializing the Field Memory. This procedure is carried out at the beginning of each survey day, (and after valid data has been dumped correctly).

To enter the operator's code (ie, to clear and initialize the field data memory), proceed as follows:

a. Turn MODE selector to TFM (for total field (3)); to GRAD (for gradiometer (5)); to AUTO (for base station (4)); or to TIE (for tie-line (3 or 5)).

b. Press one of the following keys: SPOT RECORD, MULTI RECORD or AUTO RECORD. The display will flash OPNNNN accompanied by an audible beep.

c. Press the CHANGE key; then press the following numerics, where M = 9 for alkaline batteries and any other digit for rechargeable batteries; and the Ns represent a number which identifies the operator.

3MNN for total field
4NNN for base station
5MNN for gradiometer

NOTE

1. The gradient operator code can be used for the tie-line survey.
2. If a keying error occurs when entering the numerics, press the CHANGE key again and re-enter the digits.
3. At any time that it is necessary to enter a new operator's code, the accumulated field data must be saved by dumping first. Once the new operator's code has been entered and a reading taken, recovery of the data stored prior to entry of the new operator code will not be possible.
4. The second digit of the operator code must be a 9 if alkaline batteries are used.

4.12.4 Initializing the Tie-Line Memory. This procedure is only carried out after a survey has been completed. It is required in order to enter new tie-line data for the basis of a new survey. Proceed as follows:

a. Turn the MODE selector to DUMP.

b. Press the READ key, then press the AUTO RECORD key. The display will flash HPNNNN accompanied by an audible beep.

c. Press the CHANGE key, then the digits 8888, followed by ENTER and CLEAR.

d. The tie-line data will be cleared if the OMNI IV turns off.

4.12.5 Starting the Base Station Magnetometer. Proceed as follows:

a. Set the MODE selector to AUTO.

b. Press the READ key. A reading of the total magnetic field will be taken while the displayed digits flash on and off. When the reading is completed, a beep sounds and the reading is displayed.

c. When a reading has been attempted, if the magnetometer is not tuned within the acceptable tolerance, (referred to as detuned), there will be a beeping sound and a flashing field reading with the TUNE descriptor flashing. (Refer to para 4.14.2 for the manual tuning procedure.)

d. Press the SPOT RECORD key. The base station magnetometer will beep and the display will read OPNNNN. Enter the correct operator's code (commencing with the numeric 4) as described in para 4.12.3 (ie, initializing the field memory).

CAUTION

If the data stored in the base memory of the previous day has not been dumped, the OPNNNN will not be requested. Entering an OP code will erase the data.

e. After the OP number has been entered, press SPOT RECORD again.

f. If the base station recording time interval has not been entered since memory initialization took place, it will commence at this point. The OMNI IV will beep and the previous time interval will be displayed in flashing digits. Change and enter value as necessary (between 5 seconds and 60 minutes).

g. Press SPOT RECORD again. The display will show RP001 signifying that the memory is ready to store the first reading.

h. Press SPOT RECORD again. The first reading will be stored in memory and identified as RP001 and the OMNI IV will be turned off automatically.

j. Press the READ key to start the base station again.

NOTE

From this point, the base station will function automatically. After the preprogrammed recording interval has elapsed, the OMNI IV turns itself on; displays the previous total magnetic field reading with flashing digits until the polarization sequence is complete; then, after storing the reading and incrementing the RP number, it shuts itself off. This is the cycling sequence.

4.12.6 Starting the Gradiometer. Proceed as follows:

a. Set the MODE selector to GRAD.

b. Press the READ key. The OMNI IV turns on and a total field reading is being processed while the first two displayed digits are flashing. When the reading is completed, there is a beep and the reading is displayed.

NOTE

If the OMNI IV is detuned, it will indicate this by a sequence of beeps accompanied by a flashing display of the previous reading. Refer to para 4.14.2 for the manual tuning procedure.

c. Press SPOT RECORD. This will cause the OMNI IV to beep and OPNNNN will be displayed. For the gradiometer, enter 5 followed by a 9 (if alkaline batteries are being used) or another digit if not; followed by the two operator digits.

NOTE

As soon as the new OP number is entered, the field memory will be initialized, and any stored data in memory is cleared (ie, not retrievable).

d. Press MULTI RECORD. The position number will be displayed. If correct, press MULTI RECORD again to store the data.

4.13 Pre-Survey Programming

At the base station, the following programming tasks may be accomplished with the respective MODE selectors on the base station magnetometer and total field (or gradient) magnetometer both set to TEST.

a. Clock synchronization (including TIME and DATE setting).

b. Reading synchronization (if desired).

c. For the field magnetometer (or gradiometer), programming the initial LINE, LINE SPACING, POS, and POS SPACING.

d. On the base station, the base LINE and POS readings can be entered as a reference record, then the time SPACING interval can be reprogrammed for the desired automatic recording interval cycle.

e. Then memory initialization can be completed with the entry of the OP number.

4.13.1 Real-Time Clock. The OMNI IV includes a programmable real-time clock for synchronizing readings and for recording the times of field readings. The clock also includes the date. However, this does not include leap year compensation. It is suggested that a digital wrist watch be used to set the time at the start of a survey and to synchronize the clocks within the base station magnetometer.

NOTE

If the date is changed, the time has to be re-entered as well.

To set the time, the following procedure must be followed:

a. The time can be set in any position of the MODE selector.

NOTE

Consider an example in which the display indicates the following time: 14:26:13. It is the intent to set the clock at 14:28:00. Therefore, it will be necessary to enter this set time (ie, 14:28:00) when the digital wrist watch displays 14:27:59. For convenience, the real-time clock can only be programmed for hours and minutes; not seconds. Therefore, allow at least one minute prior to the time that the entry will be made.

b. Press the CHANGE key and observe the display. Note that the numerics 1 and 3 in the seconds column have been set to zero automatically, ie, 14:26:00.

c. The display flashes on and off indicating that it is ready to accept new values. Key in the following numerics in sequence: 1, 4, 2, 8. Observe the display to confirm that the new value is entered correctly. Do not enter any digits for seconds.

NOTE

Depending upon the time difference between steps b. and c., it is most probable that the display period will time out and the audio alarm will commence beeping. To extend the time display period, press the ENTER key followed by the CHANGE key again. It may be necessary to repeat this procedure several times during the time-setting process.

d. Now monitor the wrist watch until it displays 14:27:59 then, immediately, press the ENTER key. With some experience, the setting accuracy can be less than 1 second.

e. To confirm that the real-time clock has been set properly, press the TIME key twice and compare the time on the wrist watch with the time on the display.

NOTE

Synchronization of the base station and field magnetometers requires two operators to ensure synchronization accuracies of less than one second. Every time that a reading is taken, the computer reads the real-time clock and stores the time in memory. The time at which the real-time clock is read is defined as the time immediately after the processing of the sensor signal. The same procedure is used in the base station magnetometer. This method improves the record accuracy of the actual time that the total magnetic field was measured.

To synchronize OMNI IV clocks, it will be necessary to carry out this procedure simultaneously; or to set one TIME display, then use that display as the reference to set another TIME display.

To enter or change the date:

a. Press the TIME key. The time will appear on the display.

b. Press the TIME key again immediately. The time display will convert to a date display.

c. Consider, for example, that the displayed date is 85:08:19 (ie, 19th August 1985) and that the required setting is 86:04:12 (ie, 12th April 1986).

d. Press the CHANGE key. Then, in sequence, press the digits 8, 6, 0, 4, 1, 2.

e. Verify that the date is correct, then press the ENTER key.

f. Press the TIME key two times to recall the entered date to the display.

4.13.2 Reading Synchronization. If it is required to synchronize the field unit with the base station unit, the time spacing for the field unit must be re-entered.

- a. Press the TIME key on the field unit.
- b. Press the SPACING key to display the time interval.
- c. If the time spacing is the same as in the base station unit, press the ENTER key.
- d. If the time spacing is different from the base station unit, press the CHANGE key, then enter the correct time interval.
- e. If no synchronization is required, enter a time interval of 00:00 in the field unit.

4.14 SYSTEM TUNING (Prior to Survey)

Whenever a total field measurement is to be taken for the first time, it is necessary to confirm that the predicted measurement will approximate the total field reading which has been stored previously.

If the difference between the actual total field strength and the last total field reading stored previously in memory is too large, the OMNI IV may not be capable of taking a proper reading. In such a case, the display will indicate that OMNI IV tuning is required. The TUNE descriptor will flash on and off and the audible alarm will be activated.

4.14.1 Retrieval of Last Previously Stored Total Field Value. To obtain the previous value of the total field, proceed as follows:

- a. Press the RECALL key.
- b. The display will indicate the record number of the last reading. The DECAY and SENS descriptors will flash to indicate that the unit is in recall.
- c. Press the FIELD key and observe the display. The total field value which is the last stored total field value, will be displayed.
- d. Press RECALL again to toggle back to the normal function.

4.14.2 Tuning the OMNI IV. Proceed as follows:

NOTE

As a general rule, when the difference between the local ambient field strength and the last stored total field strength is greater than $\pm 15\%$ relative to the local ambient field strength, the OMNI IV must be tuned manually.

- a. Consider the example when the display indicates a last previously stored total field strength of 50500.7 GAMMA.
- b. Furthermore, in the example, it is estimated that the local ambient field strength is 59000 γ . By calculation; $\pm 15\%$ of 59000 $\gamma = \pm 8850 \gamma$.

NOTE

The OMNI IV might not be capable of tuning automatically from 50500 to 59000 gammas. Therefore, a closer value must be inserted in the two most significant digits that are used for tuning.

- c. Press the CHANGE key. Observe the flashing total field value; 50500.7 indicating that the value may be changed now.
- d. In sequence, press the following numeric keys: 5, 9. Observe that every time a numeric key is pressed, the associated corrected digit on the display stops flashing. This procedure is equivalent to a flashing cursor which travels from left to right.
- e. After the changed numerics have been entered, confirm visually that the new entry is accurate, then press the ENTER key once. The OMNI IV is now tuned to 59 ky.

NOTE

If an incorrect numeric is entered in step d., it is not possible to cancel or edit the entry while in the process of entering. The procedure for correcting an entry is the same as for entering a new value.

- f. Assume that the numerics 5, 3 have been entered and suddenly, it is evident that a 3 has been entered erroneously instead of a 0. Press the ENTER key to enter the incorrect value. Then press the CHANGE key again and repeat step d.

4.14.3 Incomprehensible Displays. Assuming that the operator has no historical knowledge regarding the OMNI IV when the FIELD key is pressed in the TEST mode, it is possible that the display may also indicate an incomprehensible number which flashes on and off. When this occurs, the OMNI IV does not have a valid total field value in memory, (ie, the check sum for the total field is bad). In this event, the OMNI IV must be tuned to the approximate ambient local field.

- a. Carry out the tuning procedure.

4.15 SYSTEM TUNING (During a Survey)

Consider the condition where there is a warning that the OMNI IV requires tuning during an actual survey. Although this is very unlikely, it is possible if a sudden large local gradient is encountered between one station on the grid and the next station. To cause this, the local gradient must induce a change which will be larger than $\pm 15\%$ of the last stored total field strength value.

If this occurs, the total field value displayed will flash on and off, the TUNE descriptor will appear, and the audio alarm will beep.

Although there is no indication how large the gradient and the sign of the gradient is, it is suggested that the tuned value be increased by $+ 10\%$ of the last value.

The flashing total field value on the display is the last stored value which is valid. Therefore, it must be used as the reference to calculate and add $+ 10\%$.

- a. As an example, assume that the flashing total field value is 62350.3 GAMMA.
- b. By calculation, 10% of 62350 γ is approximately 6000 γ .
- c. Add 6000 γ to 62000 γ . The new tuning value shall be 68 k γ , (ie, $68 \times 1000\gamma$).
- d. Press the CHANGE key. In sequence, enter the following numeric digits: 6, 8.
- e. After the changed numerics have been entered, confirm visually that the new entry is accurate, then press the ENTER key once.

NOTE

When the OMNI IV is operated in either the GRAD or TFM mode and the OMNI IV requires tuning during an actual survey, a new reading will be taken automatically following tuning when the FIELD key is pressed. This is a feature of convenience for the operator.

- f. To obtain a reading immediately after tuning, press the FIELD key.

g. Observe the display for about 3 seconds. The display will indicate 68000.0 and the two most significant digits 6 and 8 will be flashing on and off. Flashing of the two most significant digits is confirmation that the OMNI IV is tuned to $68 \times 1000 \gamma$.

h. After this, a new total field reading will appear on the display, eg. 66100.8 GAMMA. For this example, the OMNI IV was capable of tuning to the new total field value. The OMNI IV is now tuned to 66100 γ for the next reading.

j. In the event that the OMNI IV indicates that it cannot tune even after it was tuned to 68000 γ , the operator might re-tune the OMNI IV to 72000 γ using the same procedures as outlined previously.

k. If no results are obtained after two or three attempts, tune the OMNI IV to $- 10\%$ of 62350 γ or $62000 - 6000 = 56000 \gamma$. The reason for this is that the OMNI IV may be subject to a large negative gradient relative to the local ambient field.

NOTE

In general, the operator might have to re-tune the OMNI IV several times in order to discover the new total field value. Field experience has indicated that usually the OMNI IV needs re-tuning only once.

4.15.1 Large Gradients. In the tuning procedures, it was indicated that the range of automatic tuning that the OMNI IV is capable of handling is approximately $\pm 15\%$. It must be noted that this is not an instrument fault or design limitation, but an indication of large gradient variations and this is a significant observation in the survey. One useful procedure is to monitor the DRIFT function (ie, the change in gammas between the previous total field reading and the present reading). To display the drift:

- a. Press the DRIFT key following any reading. This option remains in effect until changed by pressing the FIELD key.

NOTE

Monitoring drift makes it easy for the operator to detect large horizontal gradients, which may require additional readings (SPOT RECORDS) at intermediate fill-in stations along the survey line. The drift indication also checks data repeatability at any given station.

4.16 PREPARING THE SURVEY GRID PROFILE

A sample grid configuration is shown in Figure 3-1.

Initially, to set up the imaginary grid pattern, a definition is required regarding the direction (or ground track) that the operator must travel. Consider an example where the operator starts from a specific reference position and proceeds to the South along a specific line. See Figure 4-5. The starting position will have a reference of P 0000. If the operator proceeds initially to the South, this requires a minus (-) sign notation for the position spacing value according to the example grid. The linear units of measurement for spacing are arbitrary since they merely represent a means of identifying magnetometer measurements geographically by coordinates when the data is printed out. It is essential, however, that the spacing keyed into the OMNI IV be identical to the actual spacing established in the field survey.

As an example, consider the line spacing of 25 metres shown in Figure 4-5.

To set the initial line reference and the grid spacing between adjacent lines into the OMNI IV:

- Set the MODE selector to TEST.
- Press the READ key and observe the displayed total test field (ie, 56369.7 GAMMA).
- Press the LINE key. The actual display is irrelevant. Then press the CHANGE key. The displayed value will commence flashing on and off.
- Press the \pm key once or twice until the first two positions on the display indicate L and blank.

NOTE

The display does not indicate a + sign. Instead a blank will be observed.

- Press the 0 key four times to give a display of L 0000. This is the primary reference line.
- Press the ENTER key to enter the new value.
- Press the LINE key again. Confirm that the entry is correct.
- Press the SPACING key. The INTV descriptor will appear to indicate that this is the line spacing.
- Press the CHANGE key. The displayed value will commence flashing on and off.

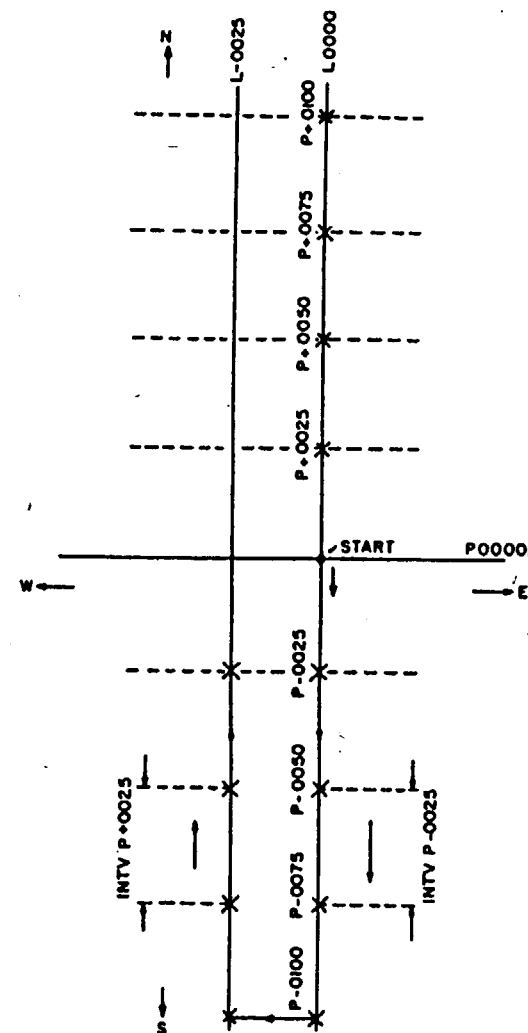


Figure 4-5 Start of Survey Profile

- k. Press the \pm key once so that the first two positions on the display indicate -.
- m. Press the following numeric keys in sequence 0, 0, 2, 5 to give a display INTV L-0025 (line spacing).
- n. Press the ENTER key to enter the new line spacing value.
- p. Press the SPACING key to recall the line spacing display. Confirm that the entry is correct.

A similar procedure applies for position (station) numbering and spacing. The position ordinates are set up using the same procedure but operating the POS key instead of the LINE key. In this example, the position spacing is also set to -0025 intervals and the zero reference is P 0000.

NOTE

To summarize the quadrant sign, tracking to the North and East are considered positive.

4.16.1 Survey Track. A typical survey track is shown in Figure 4-5. Consider the path traversed during the example survey. The origin is identified on the figure as 'start' and has the grid coordinates L 0000, P 0000.

- a. When the operator starts at the origin, if the LINE key is pressed, the value 0000 will be displayed. If the POS key is pressed, the value P 0000 will be displayed. When a reading is taken at this location, the reading must be stored in the TFM mode under MULTI RECORD key control.
- b. When the first position south of the origin is reached, a position update is required. Press the UPDATE key. The computer combines the P 0000 position entry with the INTV P-0025 spacing entry to give the new position entry of P-0025.
- c. When a total field reading is taken at this location, the position entry P-0025 is recorded together with the line entry L 0000.

NOTE

Alternatively, if the AUTO RECORD key is used, the position is updated automatically.

- d. As the survey continues Southward, this procedure decrements the position number from P-0025 to P-0050, then from P-0050 to P-0075 by using the UPDATE key, when the data is to be stored.

e. In the example, when the operator reaches P-0100, the track turns Westward to the next line, then it continues Northward. Therefore, the position number remains unchanged, but the line must be updated. When the next station Westward is reached, press the LINE key, followed by the UPDATE key. The computer combines the L 0000 line entry with the INTV L-0025 spacing entry to give the new line entry of L-0025. The position is not updated here.

f. When the total field reading is taken at this location, the entries L-0025 and P-0100 will also be recorded in the manual record mode. The reading must be stored under MULTI RECORD control because only the line number is updated.

g. The track now turns Northward, therefore the position interval must be changed from -0025 to +0025. Press the POS key, then press the SPACING key. Press the CHANGE key, then press the \pm key. Press the ENTER key. The position spacing display should read INTV P 0025.

h. As the track progresses Northward, the negative position numbers will change in increments of 0025 until P 0000 is reached. Then they will become positive, eg, P 0025, P 0050, etc.

4.17 SETTING UP A TIE LINE

For this survey method, there is no base station. Therefore, in order to utilize the data correction algorithms in the OMNI IV microprocessor, it is necessary to establish a base station (or base point) reference as well as a series of tie points (usually along the same tie line). Tie line(s) are usually located along the strike length of the survey grid, and either along the baseline or at the ends of grid lines.

NOTE

The tie-point readings form a bench mark for computing diurnal variations so that the survey readings can be corrected. Therefore, at each reading, the correct survey grid coordinates (ie, line and position) must be recorded simultaneously. Erroneous data must be deleted or readings must be repeated. Readings at the base reference point can be stored in the tie-line memory by selecting the TIE mode and entering recordings using the SPOT RECORD key. Check the reading quality display (ie, SENS and DECAY) for each reading. Discard a poor reading by pressing the OFF key. Then take a repeat reading by using the SPOT RECORD for the base reference point or the MULTI RECORD for the tie points. If the quality is poor for multiple readings, move the tie point to a location where good quality and repeatability can be obtained. The location of the base reference point should be selected in an area with low gradient.

Refer to Figure 3-2 and proceed as follows:

CAUTION

Prior to taking any reading, ensure that the sensor is oriented with its North mark (engraved on the sensor) facing North. Ensure that the sensor interconnect cable is attached securely to the OMNI IV. Keep the sensor at arm's length when taking a reading.

a. At the base reference point:

- (i) Set the MODE selector to TIE.
- (ii) Press the READ key. The display shows the total field reading at the base point.
- (iii) Press the SPOT RECORD key once. The audio beep will commence with the display flashing OPNNNN, if this is the first reading to be stored for the day.

NOTE

The first digit designates the application in which the OMNI IV was last used (eg, 3 = field magnetometer, etc).

- (iv) For the total field magnetometer application, press the CHANGE key. The beeping will stop.
- (v) Enter the OP number (ie, 3 followed by three additional digits).

NOTE

These can be any three digits but are usually assigned to identify the operator with the data stored.

- (vi) Press the ENTER key.
- (vii) Press the SPOT RECORD key. The display should show LP 9999 to indicate a base reference point.
- (viii) Press the SPOT RECORD key a second time to enter the reading into the special tie-line memory.

b. Proceed to the first tie-point position, then:

- (i) Keep the MODE selector set to TIE. Press the READ key. After the polarization, the display shows the field reading at the tie point.
- (ii) Press the MULTI RECORD key. This does not advance the tie-line number.

(iii) The display will show the tie-line number. The flashing L denotes that this is a tie-line reading.

(iv) Verify that this is the correct line number. If not, refer to para 4.16 to program the grid coordinates. Change the reading to the correct line and position number as required.

(v) Press the MULTI RECORD key again to store the reading and grid coordinates in the special tie-line memory.

c. Proceed quickly to the next tie-point position along the tie line.

(i) Press the READ key. The display should show the field reading at the previous tie point during polarization, followed by the new field reading.

(ii) Press the AUTO RECORD key. This advances the line number.

(iii) The display will show the next sequential line number. The flashing L denotes a tie-line reading.

(iv) Verify that this is the correct line number for the tie point.

(v) Press the AUTO RECORD key again to store the reading and grid coordinates in the special tie-line memory.

d. Repeat step c. along the entire length of the tie line or at the designated tie points.

CAUTION

Proceed as quickly as possible to minimize the effects of diurnal variations. If scattered tie-point locations are selected, it will be necessary to correct the line and position displays. Refer to para b. and especially step (iv).

e. When all tie points have been recorded, return to the base reference point, then:

(i) Press the READ key in the TIE mode. The display will show the total field reading at the base reference point.

(ii) Press the SPOT RECORD key. The display will show LP 9999, denoting a base reference point.

(iii) Press the SPOT RECORD key again. The reading and grid coordinates will be stored in the special tie-line memory. Therefore, there will be two readings entered for the base reference point: One at the beginning and one at the end of the tie-point reference measurements. These two readings will indicate the diurnal variation between them and the elapsed time between the two readings.

NOTE

The tie-line loop is now complete and stored in memory for the entire survey. On subsequent survey days, the first and last reading of the day must be recorded at the base reference point or at any given tie point stored in memory. These must be stored in the TIE mode.

4.17.1 Extending the Tie-Point Grid. If the survey grid is extended, it is possible to increase the number of tie-line references in the tie-line memory at any time or day during the survey. Repeat steps a. through e. at the new tie-point positions. Previous tie-line readings will remain in memory until they are erased by the HP8888 dump code. (See para 4.12.4.)

4.17.2 Displaying the Tie-Point Drift. The diurnal drift between the two most recent measurements at the tie points can be monitored as follows:

- a. Ensure that the MODE selector is set to TIE.
- b. Press the DRIFT key twice. The display will show the GRAD descriptor and the value will be the drift between the last two consecutive tie points.

CAUTION

Do not initialize the tie-line memory again until the survey is complete.

4.18 SETTING UP A LOOP SURVEY

This method is almost the same as the tie-line method except that, instead of selecting and recording numerous tie point references, only the base reference point is used as a bench mark. This method is applicable to smaller survey areas in which it is practical to return to the base reference point frequently to take subsequent readings. The looping survey diagram is shown in Figure 3-3.

CAUTION

Before the looping method can be used, the tie-line memory must be initialized. (See para 4.12.4.) Then the base reference point only will be stored. If it is not initialized, existing tie-point information will be picked up and used in computations.

4.19 FIELD MEASUREMENTS

If this is the first day of the survey, the OP number will not have been programmed. On subsequent days, the OP number will have to be programmed at the start of each day after the survey data from the previous day has been dumped.

NOTE

When measuring the total field (TFM mode), use the OP3NNN number. When measuring gradient (GRAD mode), use the OP5NNN number.

CAUTION

At each station, check the reading quality and repeatability by observing the DELAY and SENS displays. Also monitor the drift between consecutive stations. Discard any bad or doubtful readings by pressing the OFF key. Then take a new reading.

To take field measurements, proceed as follows:

CAUTION

It is not possible to correct erroneous coordinates once the data is stored in memory except by erasing the previous reading. (See para 4.20.) This also applies to computations which utilize values obtained from an address identified by LINE and POS coordinates.

- a. Upon arriving at a station, check the surveyed line and station number and compare them with the LINE and POS numbers programmed into the OMNI IV.
- b. Position the sensor correctly. Set the MODE selector to TFM.
- c. Press the READ key. The previous total field reading will appear in flashing digits while the reading cycle is being completed.

NOTE

If the reading cycle has been synchronized with the base station unit, (ie, the readings occur at the same time that the base station takes a reading), a count-down to the next read cycle will appear first. (See para 4.13.2.)

- d. Press the MULTI RECORD key. The display shows the position number. Verify that this position or station number is correct for where the reading is to be stored. Then press the LINE key. Verify the correct line number. (If not, reprogram these values.) If the line and position numbers are correct, press the MULTI RECORD key twice to store the first total field reading of the survey.

e. Proceed to the next survey station.

f. Press the READ key. The flashing display shows the previous total field reading until the new reading cycle is completed. Then it shows the reading for the current location.

g. Press the AUTO RECORD key. The display shows the updated station number.

h. Press the AUTO RECORD key again to store the new data.

j. Repeat steps e. through h. at each position along the survey grid line.

NOTE

If the line and position of a survey location correspond to the tie-line point, the P or P- on the display will flash to indicate a tie-point station.

k. When the end of a survey grid line is reached, it is necessary to transfer to the next line without changing the position number, then to change the spacing sign. Refer to para 4.16.1 for this procedure.

4.20 Erasing Field Data Records

To erase data from memory, one record at a time, proceed as follows:

a. To erase tie-line readings, set the MODE selector to TIE. To erase field readings, set to TFM, or to GRAD (for readings in gradiometer mode).

b. Press the READ key and wait 2 seconds until the cycle is complete.

c. Press the RECALL key, then check the field, time, line or pos to verify that this is the reading to be erased.

d. Press the CLEAR key.

e. Repeat the sequence in a. through d.

NOTE

The block erase feature works in the data-dump mode also. Similarly, it is necessary to select RECALL.

4.21 Choosing Recording Format

Depending upon the character of the survey grid and the survey conditions, the operator can choose from three recording formats.

a. SPOT RECORD. This key is used where grid coordinates are not required. The data block is identified by an RP number. Do not use this key in the TIE mode as this will indicate a base reference station.

(i) Press the READ key. The display shows the total field reading.

(ii) Press the SPOT RECORD key once. The display assigns the next sequential record number (RP) to the reading.

(iii) Press the SPOT RECORD key again. The reading is stored in the memory data block.

b. MULTI RECORD. This key is used to store many readings at the same position (station) number.

(i) Press the READ key. The display shows the total field reading.

(ii) Press the MULTI RECORD key once. The display shows the previous position number.

(iii) Press the MULTI RECORD key again. The reading is stored in the memory data block.

c. AUTO RECORD. This key is used to update the position (station) number at pre-programmed spacing increments.

(i) Press the READ key. The display shows the total field reading.

(ii) Press the AUTO RECORD key once. The display updates the position (station) number to the next position by adding (or subtracting) the spacing interval.

(iii) Press the AUTO RECORD key again. The reading is stored in the memory data block.

4.22 Skipping Stations

Where it is not possible to collect data at a given position (eg. because of water or topographical obstruction), the position (station) number can be updated without storing a field reading. Proceed as follows:

a. Proceed around obstruction to where a field reading can be taken.

b. Press the READ key. The total field reading will be displayed.

c. Press the POS key.

d. Press the UPDATE key once, or for as many times needed to arrive at the new position number.

e. At the correct position number, press the MULTI RECORD key twice to store the reading in the memory data block.

4.23 Intermediate Points

On a survey grid with 25 m spacing increments, it is desirable to record data at the mid points (eg, 12.5 m, 37.5 m, etc). The display is only capable of indicating the two digits, therefore, when a space of 12.5 m is required, only the digits 12 are entered. Then the display shows 00:12 with the colon and decimal point together and flashing. This readout actually means 12.5 m even though the .5 is not displayed.

SECTION 5

DATA RETRIEVAL

5.1 RETRIEVAL CONCEPTS

Data stored in the OMNI IV memories can be processed and dumped using various program directives. These directives (access codes) are entered through the keypad.

The data is dumped by connecting the OMNI IV to an applicable peripheral device such as a printer or computer. The connection is made through a cable which is attached to the connector on the rear of the console. (When the OMNI IV is chest mounted, the data dump connector is on the underside of the console adjacent to the operator's right hand.)

5.1.1 Diurnal Correction of Data. When the OMNI IV is used in the tie-line or looping mode, the data stored in the special tie-line memory file is merged with the field measurements stored in the field data memory file. The internal microprocessor then computes the diurnal variation in the earth's magnetic field for each survey grid coordinate and corrects the field data file using the tie-line file. No manual computations are required. This correction process takes place during the data dump.

In addition to the self-correcting feature for data using the tie-line or looping method, the OMNI IV can also include a base station (or autocycling) option. In this method, the base station records the total field reading at a single location but at precise time intervals. During the data dump, the raw data from the field magnetometer is dumped to the base station magnetometer where it is merged. The internal microprocessor then computes the diurnal variation in the earth's magnetic field for each survey grid coordinate by comparing the times at which readings were taken and computing any mid-interval values. This is most useful in Northern latitudes where more detailed monitoring of the diurnal variations is required. This correction process takes place during the data dump.

Another correction which can be applied during the data dump is the programmable datum or the programmable reference field. In this application, the OMNI IV is programmed to remove a designated datum (or value) from the field measurements. Removal of this coarse background value facilitates plotting and interpretation of data.

5.1.2 Data Dump Configurations. There are basically two equipment interconnection configurations:

a. The OMNI IV can be connected directly to the printer or computer.

b. An OMNI IV field magnetometer can be connected to an OMNI IV base station magnetometer and then to a printer or computer.

NOTE

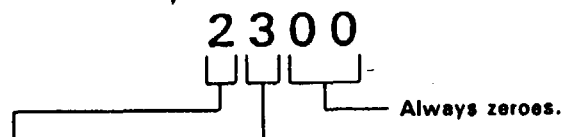
In order to hook up an OMNI IV to a computer, the user must be able to control the DTR (data terminal ready) line and the CTS (clear to send) line on that computer.

5.1.3 Interface Cables. EDA produces a series of optional data cables for interconnecting the OMNI IV to various printers and computers. The list is given in Table 2-1. Each data cable includes a communications buffer board custom designed to suit the interface requirements. See Figures 5-5 thru 5-10.

5.2 Dump Set-Up

Using the applicable interface cable interconnect the OMNI IV to the printer or computer. (Computer handshaking procedures are beyond the scope of this manual.)

5.2.1 Access Code. Data stored in memory in the OMNI IV is retrieved by entering a four-digit access code. The composition of the code is as follows:



First Digit: Type of data to be retrieved.

1 = Raw data dump. The contents in the memory are dumped directly to the recording device.

2 = Corrected data where:

a. The OMNI IV can correct its own total field data when used as a tie-line magnetometer.

b. The OMNI IV is used as a base station to read raw data from the field magnetometer, then to correct it by linear interpolation.

3 = Verification. The field data is dumped through the base station without correction being applied. Rerouting of the data dump cable is not required.

4 = Tie-point drift. The diurnal drift for the individual tie points is retrieved by subtracting the programmable reference field.

5 = Corrected tie-line data. The tie-line data file is dumped directly.

Second Digit: Formats the line number with a compass bearing.

0 = Standard +/- data presentation.

1 = North (+) or South (-).

2 = East (+) or West (-).

3 = This is a fixed format for additional computer processing. It is operable only in PROG mode.

5.3 DATA DUMP PROCEDURES

Proceed as follows:

- Interconnect the OMNI IV(s) with the applicable peripheral device.
- Carry out the appropriate procedures related to the peripheral device.

5.3.1 Initiating a Raw Data Dump. Figures 5-1 and 5-2 show sample print-outs of uncorrected total field data and base station data. Figure 5-3 shows tie-point drift and tie-line data. Proceed as follows:

- Set the MODE selector to DUMP.
- Press the READ key. The serial number of the OMNI IV will appear on the display.
- Press the AUTO RECORD key. The display will indicate flashing digits HPNNNN together with audible beeping.
- Press the CHANGE key, then enter the required access code:
 - Key in the number 1 to dump the raw data.
 - Key in a digit (0 thru 3) depicting the desired format for the line direction.
 - Key in 0, 0 if required.
 - Press the ENTER key.
- Power up the peripheral device.
- Press the AUTO RECORD key to begin the new data transfer.

```

EUA OMNI IV "Tie-line" Mag Ser #14015
TOTAL FIELD DATA (uncorrected)
Date: 4 AUG 83
Operator: 5012
Datum Subtracted: 0.0
Batt: 15.4 Volt Lithium Batt: 3.3 Volt
Last time update: 1 AUG 83 15:22:00
Start of print: 4 AUG 83 18:29:43

LINE 54+00 N DATE 4 AUG 83
POSITION FIELD ERR DRIFT TIME #1 DS
31+00 7508.4 .10 0.0 15:00:35 88
31+50 7504.3 .08 0.0 15:00:43 88
32+00 7505.0 .08 0.0 15:00:47 88
32+50 7501.9 .10 0.0 15:00:51 88
33+00 7504.1 .08 0.0 15:00:54 88
33+50 7503.1 .07 0.0 15:01:00 88
34+00 7511.1 .07 0.0 15:01:03 88
34+50 7514.9 .09 0.0 15:01:09 88
35+00 7511.1 .08 0.0 15:01:17 88
    
```

Figure 5-1 Uncorrected Total Field Data

```

EUA OMNI IV "Tie-line" Mag Ser #31005
BASE STATION DATA
Date: 4 AUG 83
Operator: 4007
Reference Field: 57500.0
Line 20+00 N Position 30+25 E
Batt: 12.1 Volt Lithium Batt: 3.2 Volt
Last time update: 1 AUG 83 15:22:00
Start of print: 4 AUG 83 18:59:53

```

TIME	FIELD	CHANGE	DRIFT	RECORD
15:00:33	57517.8	0.4	17.8	1
15:00:38	57508.6	-9.2	8.6	2
15:00:43	57511.7	3.1	11.7	3
15:00:48	57510.5	-1.2	10.5	4
15:00:53	57518.0	7.5	18.0	5
15:00:58	57523.9	7.9	23.9	6
15:01:03	57514.9	-11.1	14.9	7
15:01:08	57508.4	-6.5	8.4	8
15:01:13	57512.9	4.5	12.9	9
15:01:18	57512.7	-0.2	12.7	10

Figure 5-2 Base Station Data

```

EUA OMNI IV "Tie-line" Mag Ser #14015
TOTAL FIELD DATA (uncorrected)
Date: 4 AUG 83
Operator: 5012
Datum Subtracted: 0.0
Batt: 15.6 Volt Lithium Batt: 3.3 Volt
Last time update: 1 AUG 83 15:22:00
Start of print: 4 AUG 83 18:29:43

```

LINE	54+00 N	DATE	4 AUG 83	TIME	DS
31+00	57508.4	.10	0.0	15:00:35	88
31+30	57506.3	.08	0.0	15:00:43	88
32+00	57505.0	.08	0.0	15:00:47	88
32+30	57501.9	.10	0.0	15:00:51	88
33+00	57504.1	.08	0.0	15:00:54	88
33+30	57503.1	.07	0.0	15:01:00	88
34+00	57511.1	.07	0.0	15:01:05	88
34+30	57514.9	.09	0.0	15:01:09	88
35+00	57511.1	.08	0.0	15:01:17	88

Total Field Data (Uncorrected)

Figure 5-3 Tie-Point Drift and Tie-Line Data

5.3.2 Correcting Total Field Data. Figure 5-4 shows a sample print-out of OMNI IV corrected total field data. To correct total field data using the tie-line or looping method, proceed as follows:

- Set the MODE selector to DUMP.
- Press the READ key. The serial number of the OMNI IV will appear on the display.
- Press the AUTO RECORD key. The display will indicate flashing digits HPNNNN together with audible beeping.
- Press the CHANGE key, then enter the required access code:
 - Key in the number 2 to dump corrected data.
 - Key in a digit (0 thru 3) depicting the desired format for the line direction.
 - Key in 0, 0.
 - Press the ENTER key.
- Power up the peripheral device.
- Press the AUTO RECORD key. The tie-line memory data is then merged with the field data and self correction begins.

```

EUA OMNI IV "Tie-line" Mag Ser #14015
TOTAL FIELD DATA (base stn. corrected)
Date: 4 AUG 83
Operator: 5012
Datum Subtracted: 57000.0
Batt: 15.6 Volt Lithium Batt: 3.3 Volt
Last time update: 1 AUG 83 15:22:00
Start of print: 4 AUG 83 18:39:53

```

```

BASE STATION Ser #31005
Date: 4 AUG 84
Operator: 4012
Reference Field: 57500.0
Line 20+00 N Position 30+25 E
Batt: 12.1 Volt Lithium Batt: 3.2 Volt
Last time update: 1 AUG 83 15:22:00
Start of print: 4 AUG 83 18:39:53

```

LINE	54+00 N	DATE	4 AUG 83	TIME	DS
31+00	494.3	.10	14.1	15:00:35	88
31+30	494.6	.08	11.7	15:00:43	88
32+00	494.3	.08	10.7	15:00:47	88
32+30	486.9	.10	15.0	15:00:51	88
33+00	481.4	.08	22.7	15:00:54	88
33+30	481.6	.07	21.5	15:01:00	88
34+00	498.9	.07	12.2	15:01:05	88
34+30	505.6	.09	9.3	15:01:09	88
35+00	498.4	.08	12.7	15:01:17	88

Figure 5-4 OMNI IV Corrected Total Field Data

5.3.3 Correcting Total Field Data Using a Base Station. Figure 5-2 shows a sample print-out of base station data.

On both the base station OMNI IV and the field OMNI IV, proceed as follows:

- a. Set the MODE selector to DUMP.
- b. Press the READ key. The serial number of the OMNI IV will appear on the display.
- c. Press the AUTO RECORD key. The display will indicate flashing digits HPNNNN together with audible beeping.

On the OMNI IV used as a field magnetometer or gradiometer:

- a. Press the CHANGE key, then enter the required access code:
 - (i) Key in the number 1 to send raw field data to the base station unit.
 - (ii) Key in a digit (0 thru 3) depicting the desired format for the line direction.
 - (iii) Key in 0, 0 if digits were not 0, 0.
 - (iv) Press the ENTER key.

On the OMNI IV used as a base station:

- a. Press the CHANGE key, then enter the required access code:
 - (i) Key in the number 2 to correct the field unit data.
 - (ii) Key in the same second digit as in a.(ii) above.
 - (iii) Key in 0, 0 if required.
 - (iv) Press the ENTER key.

For both OMNI IV units:

- a. Power up the peripheral device.
- b. Press both AUTO RECORD keys simultaneously. Data transfer will commence and data correction will occur in the base station OMNI IV.

5.3.4 Verification of Raw Field Data. On both the base station OMNI IV and the field OMNI IV, proceed as follows:

- a. Set the MODE selector to DUMP.
- b. Press the READ key. The serial number of the OMNI IV will appear on the display.
- c. Press the AUTO RECORD key. The display will indicate flashing digits HPNNNN together with audible beeping.

On the OMNI IV used as a field magnetometer or gradiometer:

- a. Press the CHANGE key, then enter the required access code:
 - (i) Key in the number 1 to dump raw field data.
 - (ii) Key in a digit (0 thru 3) depicting the desired format for the line direction.
 - (iii) Key in 0, 0.
 - (iv) Press the ENTER key.

On the OMNI IV used as a base station:

- a. Press the CHANGE key, then enter the required access code:
 - (i) Key in the number 3 to verify the field unit data.
 - (ii) Key in the same second digit as in a.(ii) above.
 - (iii) Key in 0, 0.
 - (iv) Press the ENTER key.

For both OMNI IV units:

- a. Power up the peripheral device.
- b. Press both AUTO RECORD keys simultaneously. Data transfer will commence and data correction will occur in the base station OMNI IV.

5.3.5 Acquisition of Drift Measurements Between Tie Points. To dump the diurnal drift for the tie-point measurements in the tie-line unit, proceed as follows:

- a. Set the MODE selector to DUMP.

b. Press the READ key. The serial number of the OMNI IV will appear on the display.

c. Press the AUTO RECORD key. The display will indicate flashing digits HPNNNN together with audible beeping.

d. Press the CHANGE key, then enter the required access code:

- (i) Key in the number 4 to dump the tie-point drift.
- (ii) Key in a digit (0 thru 3) depicting the desired format for the line direction.
- (iii) Key in 0, 0.
- (iv) Press the ENTER key.

e. Power up the peripheral device.

f. Press the AUTO RECORD key to begin the transfer of tie-point drift data. An 'R' denotes that the diurnal drift calculation is derived from a base reference point reading; a 'T' denotes that the drift correction is derived from a tie-point measurement.

5.3.6 Retrieval of Corrected Tie-Line Data. On the OMNI IV used as a tie-line field magnetometer or gradiometer;

a. Set the MODE selector to DUMP.

b. Press the READ key. The serial number of the OMNI IV will appear on the display.

c. Press the AUTO RECORD key. The display will indicate flashing digits HPNNNN together with audible beeping.

d. Press the CHANGE key, then enter the required access code:

- (i) Key in the number 5 to dump corrected tie-line data.
- (ii) Key in a digit (0 thru 3) depicting the desired format for the line direction.
- (iii) Key in 0, 0.
- (iv) Press the ENTER key.

e. Power up the peripheral device.

f. Press the AUTO RECORD key. Data transfer will commence and data correction will occur in the tie-line OMNI IV.

5.3.7 Programmable Datum. By first subtracting a coarse field value from each field measurement collected on a survey grid, it is considerably more accurate to plot the differences between readings. (For example, if adjacent readings of 57,500 and 57,700 gammas are recorded, then a coarse value of 57,000 is subtracted from each, the resultant plot would be values of 500 and 700.) To remove the preset datum from the field data, proceed as follows:

a. Set the MODE selector to DUMP.

b. Press the READ key. The serial number of the OMNI IV will appear on the display.

c. Press the ERROR key twice (ie, DATUM is activated). If datum displayed is not as required, press the CHANGE key, then key in numerics to construct desired datum.

d. Press the ENTER key.

e. Proceed with any one of the data dump procedures in paras 5.3.1 through 5.3.4.

5.3.8 Programmable Reference. By first subtracting a coarse field value from each base station measurement collected on a time-interval basis, it is considerably more accurate to plot the drift in the magnetic field. To remove the preset reference field, proceed as follows:

a. Set the MODE selector to DUMP.

b. Press the READ key. The serial number of the OMNI IV will appear on the display.

c. Press the REF.FLD key once. If the reference field datum displayed is not as required, press the CHANGE key, then key in the numerics to construct the desired reference value.

d. Press the ENTER key.

e. Proceed with the data dump from the base station or tie-line unit using the procedure in para 5.3.1 or 5.3.6.

5.3.9 Field Dumping of Data. It may be necessary to dump unwanted data during a field survey without having a printer connected so that the field data memory can be initialized (ie, entering the OP number). Proceed as follows:

a. Set the MODE selector to DUMP.

b. Press the READ key, then press the AUTO RECORD key. The display shows a flashing number HPNNNN.

c. Press the CHANGE key, then key in 1000. Press ENTER.

d. Press the AUTO RECORD key. No key will respond because the unit is waiting for a ready signal from the printer.

e. Press the OFF key.

5.4 Profile Plots

Profile plots can be developed using the data collected by the OMNI IV. Choose any printer with a width between 40 and 190 characters. If the printer width is 70 characters or less, only the profile plot is printed. Printers wider than 70 characters can accommodate the profile plot and the data. When the paper width is less than 70 characters, the data can be printed out in digital format first using the normal data dump procedures. Then the plot can be performed using the PROG mode. The paper plot can be performed using the PROG mode. The paper strips can be matched side by side to give the overall format. A sample profile plot showing total field and gradient is given in Figure 5-5. Proceed as follows:

a. Interconnect the line printer and turn on the power.

b. Set the MODE selector to PROG.

c. Press the READ key. The serial number of the OMNI IV will appear on the display.

d. Press the AUTO RECORD key. The display will show 1PNNNN. This denotes the first parameter which is the printer paper width to be used. If the parameter is correct, press ENTER.

e. If the parameter requires changing, press the CHANGE key, then key in the new parameter. (For example, for a 40 character printer, key in 0040; for 190 characters, 0190.) Press ENTER. The printer will verify that the parameter is entered.

f. Press the AUTO RECORD key again. The display will show 2PNNNN. This denotes the second parameter which is the total field full scale shown as X on the print-out. If the parameter is correct, press ENTER.

g. If the parameter requires changing, press the CHANGE key, then key in the new parameter. (The largest field scale is 9999 gammas.) Press ENTER. The printer will verify the new full scale.

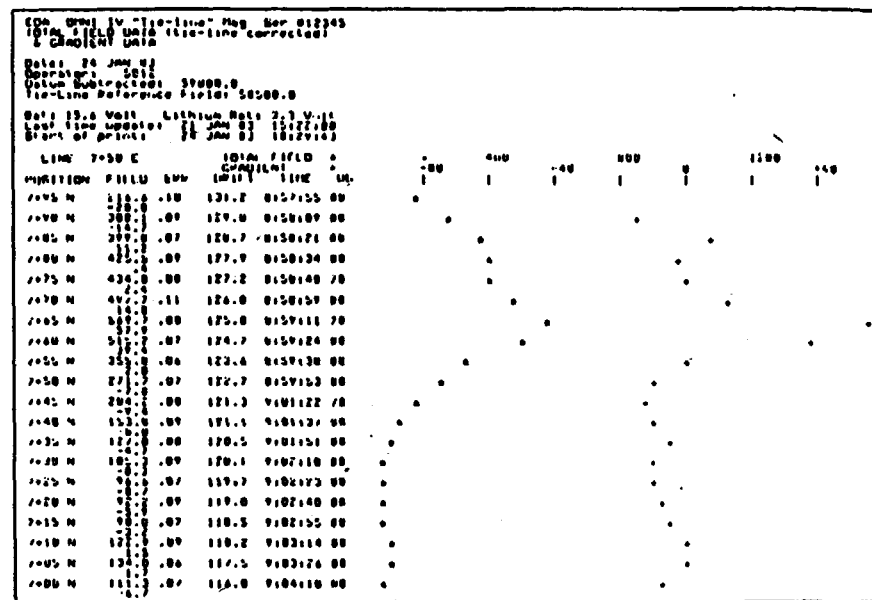


Figure 5-5 Profile Plot of Total Field and Gradient

h. Press the AUTO RECORD key again. The display will show 3PNNNN. This denotes the third parameter which represents the full scale of the gradient data being presented.

i. If the OMNI IV does not have a gradient capability, press the CHANGE key, then key in 0000. Press ENTER.

k. If the OMNI IV has gradient capability and the parameter is correct, press ENTER. If the parameter requires changing, press the CHANGE key, then enter the new parameter. (The largest field scale is 9999 gammas.) Press ENTER. The printer will verify the new full scale.

NOTE

If the gradient scale is set to 3P0000, a fine total field scale (denoted by +) will also be plotted at a 10 times finer scale than the total field scale (ie, 2PNNNN + 10, full scale).

CAUTION

If any parameter has been entered incorrectly, all three entries must be repeated.

m. After all three parameters have been entered, press the AUTO RECORD key to start the profile plot of data.

NOTE

If the paper width is less than 70 characters, the data blocks can be dumped first, then the profile plot underneath it. Then the two printed outputs can be placed side by side for the overall picture.

5.5 Specifications of the ASCII Fixed Format CPU Dump

In the ASCII CPU dump, the fields are rigidly defined.

First, there are a few empty lines with: 'cr,lf,nul,nul'. All the lines with data start with a three character alphanumeric field, not belonging to the data, but defining what type of data/header it is:

'H0' first line of header data.
'H1' second line of header data.
'H2' third line of header data.
'H3' fourth line of header data.
'H4' fifth line of header data.

'D' magnetometer data, preceding each line of measurement data.

DATA FORMAT FOR FIELDMAG or GRADIOMETER DATA (corrected and uncorrected). (BASE-STATION (uncorrected only) DATA EXITS OUT OF THE FIRST 26 FIELDS, including 'C' for the checksum.

```

1      2      3      4      5      6
123456789012345678901234567890123456789012345678901
D 1107080114 561234 1123* 561234 42000 -55125 250078 561234
xxxMMDDHHMMSSFFFFFFRRRRRCBBBBBLLLLLLLLPPPPPP EEEEDSGCGCGG
```

Format	Column	Description
xxx	1-3	3 character alphanumeric preamble for measurement data (always 'D').
MMDDHHMMSS	4-13	10 character numeric field for date and time.
FFFFFF	14-20	7 character numeric field for raw upper sensor data, with implied decimal point before the last digit, ie, 56123.4 gamma.
RRRRR	21-25	5 character numeric field for the reading number (or record pointer 'RPxxxx' on display).
C	26	1 alphanumeric character checksum error (blank if all OK).
BBBBBBB	27-33	7 character numeric field for corresponding base station reading to be used for diurnal correction.
LLLLLLL	34-40	7 character numeric field for line number with implied decimal point before the last digit, and preceded by a '-' sign if either S or W '-121250' = 121S + 25 or 12125 S.
PPPPPP	41-47	7 character signed numeric field for station or position number in the same way as the LINE number.
EEEE	49-52	4 character numeric field for error with implied decimal point after the second digit, ie, xx.xx.
D	53	1 character numeric field for the decay rate (1 to 8).
S	54	1 character numeric field for the sensor strength (1 to 8).
GGGGGGG	55-61	7 character numeric field for the raw field strength of the bottom sensor with implied decimal point before the last digit. This value minus the upper sensor strength gives the gradient.

Examples:

```

D 1107080114 561234 1123* 561234 42000 -55125 250078 561234
D 813140136109500012300 1011234 -42000 35250 7881084000
```

NOTE

A blank should be regarded as a zero. The whole string has leading zero blanking.

Header Formats:

'H0 IISSSSSXXYYRRRRRRROOOHHhhMMMMVVVV'

'H0' 3 character alphanumeric field for first header line.
 II 2 character numeric field for instrument code.
 SSSSSS 6 character numeric field for serial number.
 XX 2 character numeric field (00 if uncorrected, 99 if corrected).
 YY 2 character numeric field (internal record length).
 RRRRRR 6 character numeric field for the number of records or readings.
 0000 4 character numeric field for the operator number.
 HH 2 character numeric field for HP-CODE used in field mag.
 hh 2 character numeric field for HP-CODE used in base station (correcting mag).
 MMMM 4 character numeric field for main battery voltage.
 VVVV 4 character numeric field for lithium battery voltage.

'H1 PPPPPPLLLLLLRRRRRRRDDDDDD'

'H1' 3 character alphanumeric preamble for 2nd header line.
 PPPPPP 7 character numeric field for position or station of base mag (if used).
 LLLLLL 7 character numeric field for line of base station mag (if used).
 RRRRRR 7 character numeric field for the base-reference field.
 DDDDDD 7 character numeric field for the datum field of grid.

'H2 MMDDHHMMSSmddhmmssMMDDHHMMSS'

'H2' 3 character alphanumeric preamble for 2nd header line.
 MMDDHHMMSS 10 character numeric field for time of field mag time update.
 mddhmmss 10 character numeric field for time of first reading of survey.
 MMDDHHMMSS 10 character numeric field for time of data dump. All times are in month/day, hour:minute:seconds format.

H3 3 character alphanumeric preamble for 3rd header line.

All data fields are the same as for 'H2', except that the times apply to the base station mag use in the correction mode (if applicable).

H4 3 character alphanumeric preamble for 4th header line.

No data fields presently specified (for future expansion only).

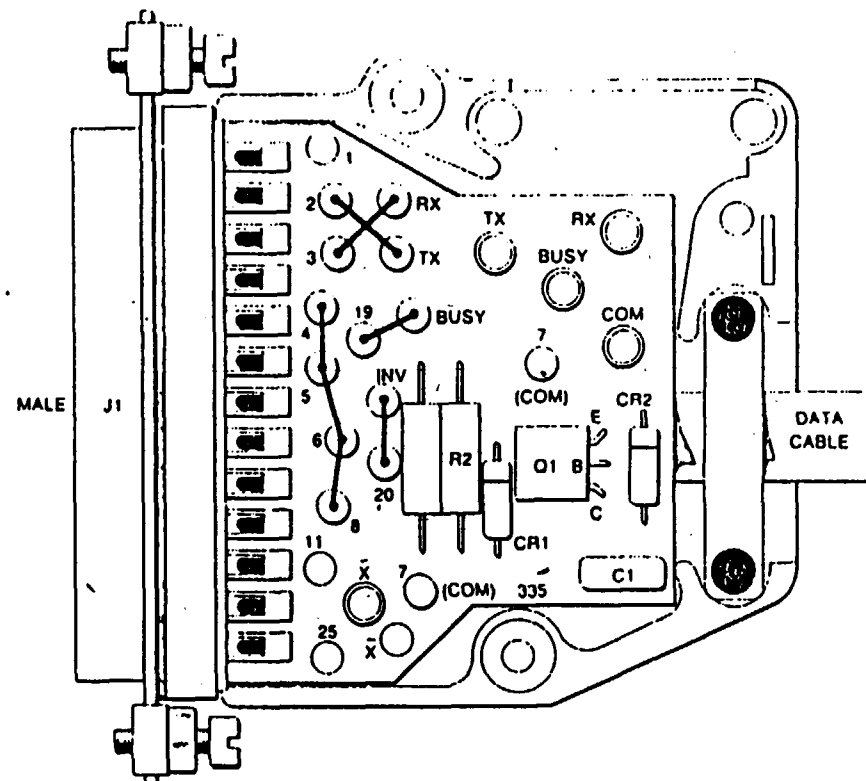


Figure 5-6 Interconnect Cable (Data Correction to TRS80/100), Male, (1-020-00335)

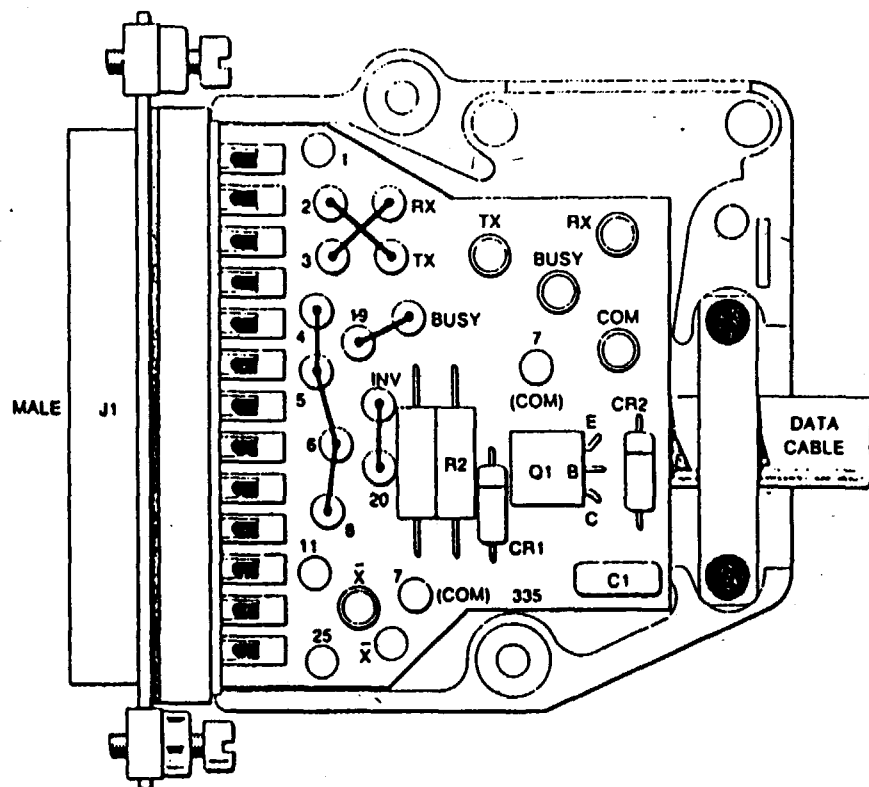


Figure 5-7 Interconnect Cable (Data Correction to Printer),
Male, (1-020-00335) DCX-443

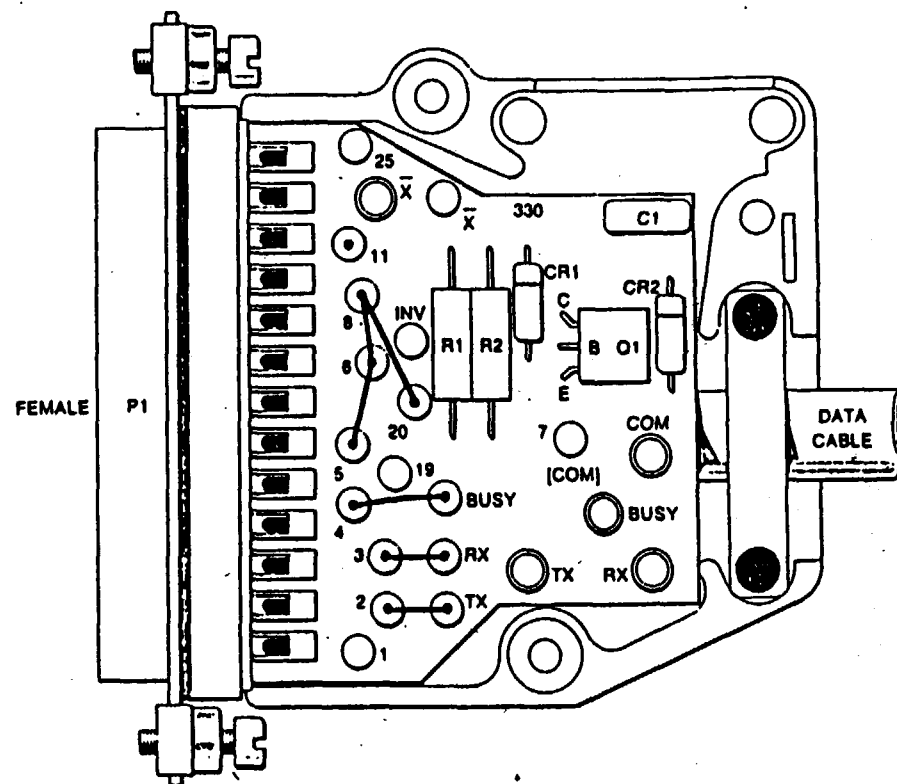


Figure 5-8 Interconnect Cable (Data Correction to Heathkit H-14 Printer),
Female, (1-020-00330)

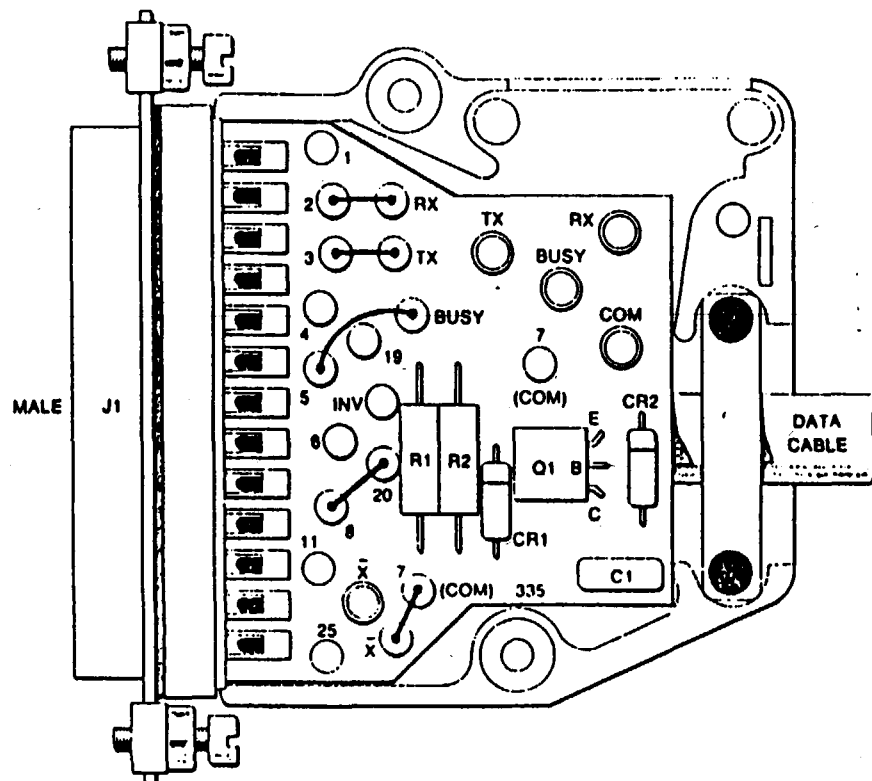


Figure 5-9 Interconnect Cable (Data Correction to HP-85 Option 0),
Male, (1-020-00335) DCX-446

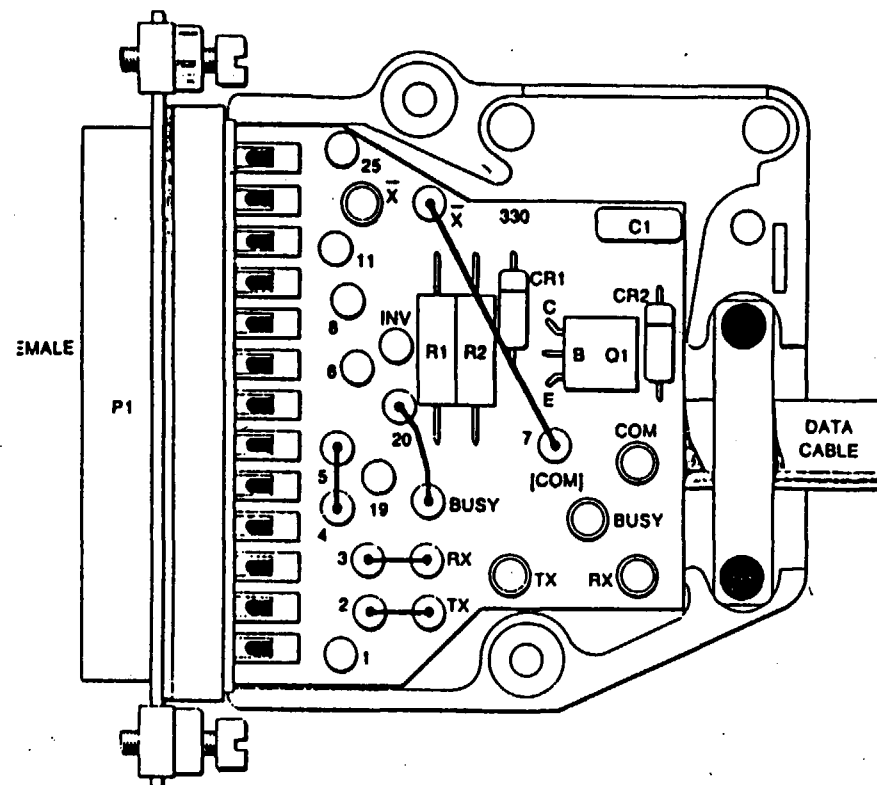


Figure 5-10 Interconnect Cable (Data Correction to HP-85 Option 1),
Female, (1-020-00330) DCX-446

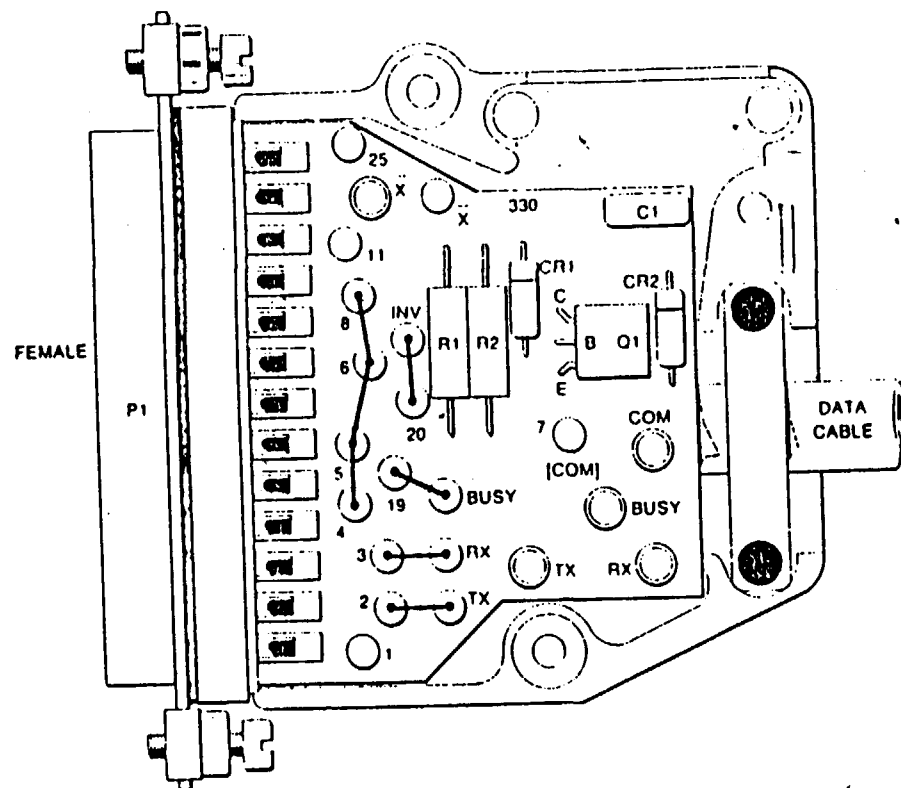


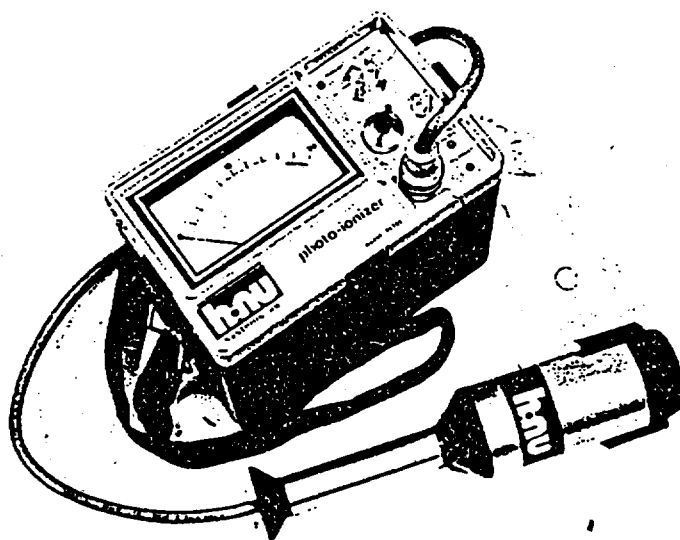
Figure 5-11 Interconnect Cable (Data Correction to IBM PC),
Female, (1-020-00330) DCX-447

Appendix A-A4

Calibration and Maintenance of HNu Photoionization Detector (PID)



INSTRUCTION MANUAL
FOR
MODEL PI 101
PHOTOIONIZATION ANALYZER



SECTION 2

OPERATION

2.1 Unpacking

Unpack the instrument carefully and remove the housing, the probe and any spare parts from the shipping carton. Place the instrument on a table or bench with the label upright. Remove the top section of the instrument by opening the two fasteners on the cover (see figure 1). The inner panel of the top section can be removed by pulling up on the fasteners. The top section of the instrument contains the battery charger and a waist strap. The waist strap clips on to the strap brackets of the instrument when needed.

Before attaching the probe, check the function switch on the control panel to make sure it is in the off position. The 12 pin interface connector for the probe is located just below the span adjustment on the face of the instrument (see figure 2). Carefully match the Alignment Key in the probe connector to the 12 pin connector on the control panel, and then twist the probe connector until a distinct snap and lock is felt.

Attached to the instrument is a warranty card which should be filled out completely and returned to HNU Systems.

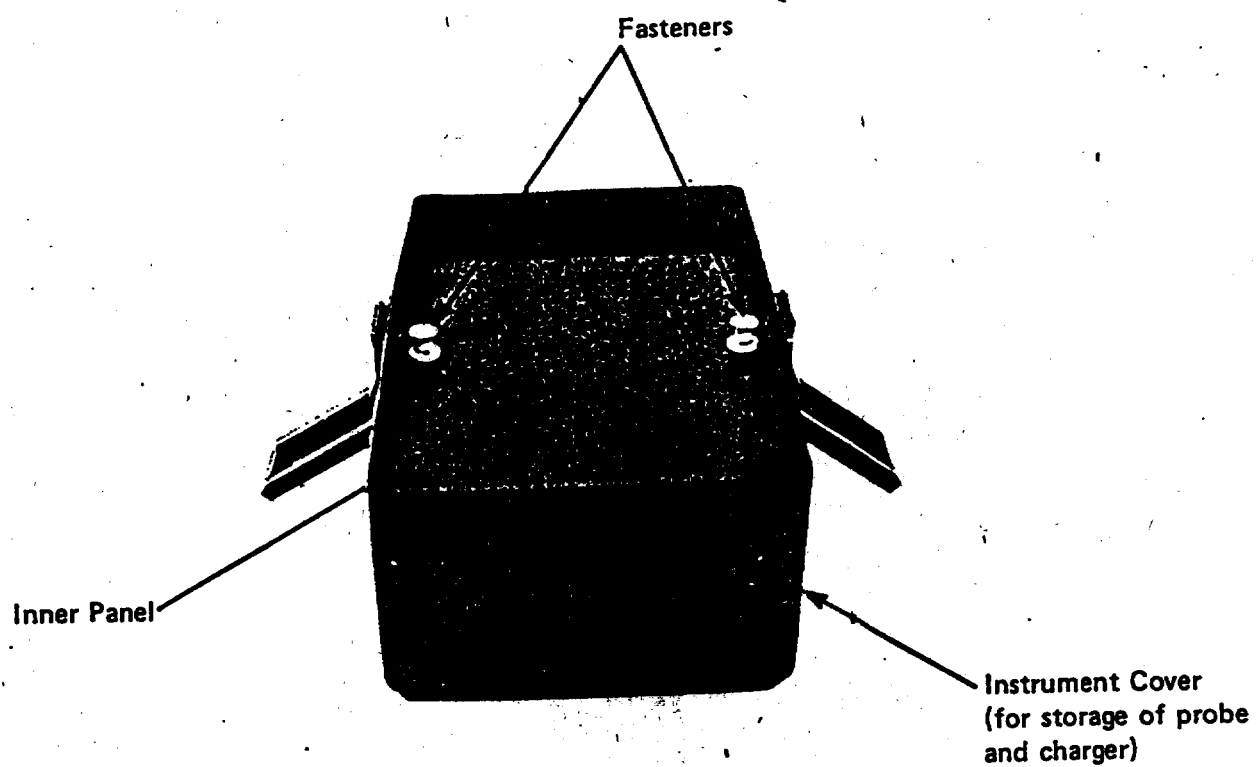
2.2 Operation

Turn the function switch to the battery check position. The needle on the meter should read within or above the green battery arc on the scaleplate. If the needle is in the lower portion of the battery arc, the instrument should be recharged prior to making any measurements. If red LED comes on, the battery should be recharged.

Next, turn the function switch to the on position. In this position the UV light source should be on. Look into the end of the probe to see the purple glow of the lamp.

A brief description of the instrument controls and functions is shown in Figure 2.

Figure 1 (Continued)



Top View

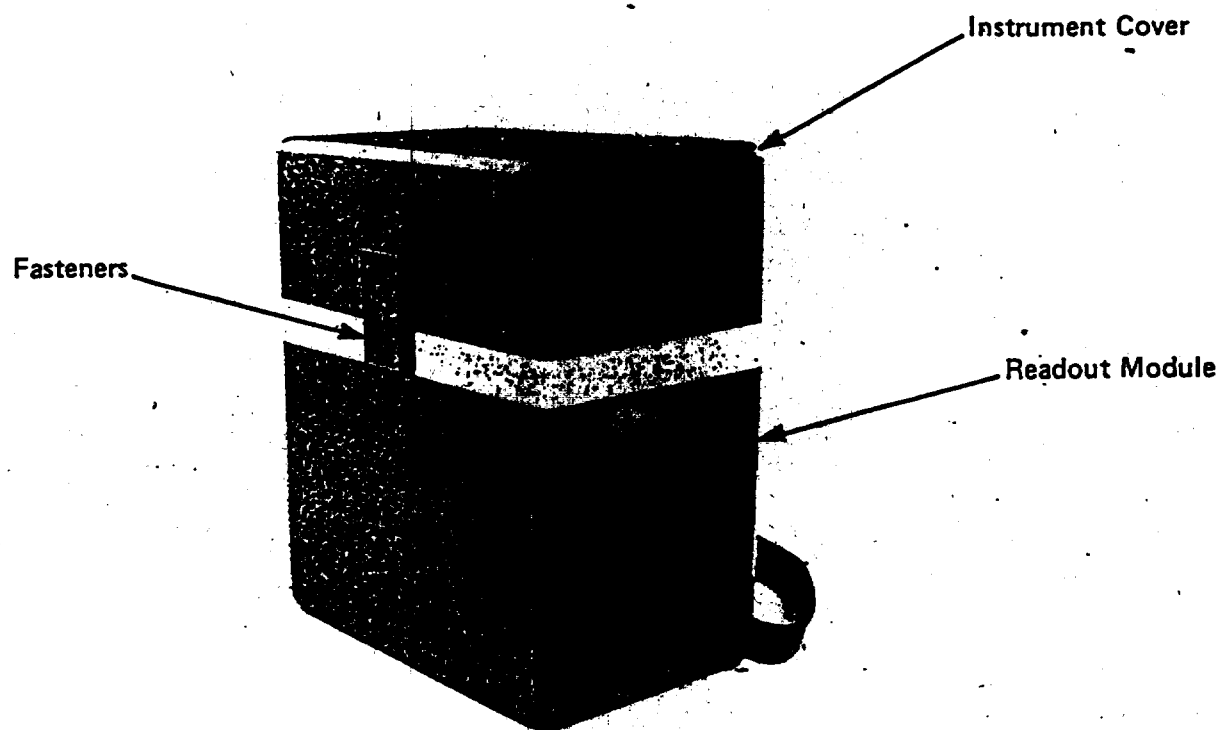


Figure 1. Unpacking the Photoionizer.

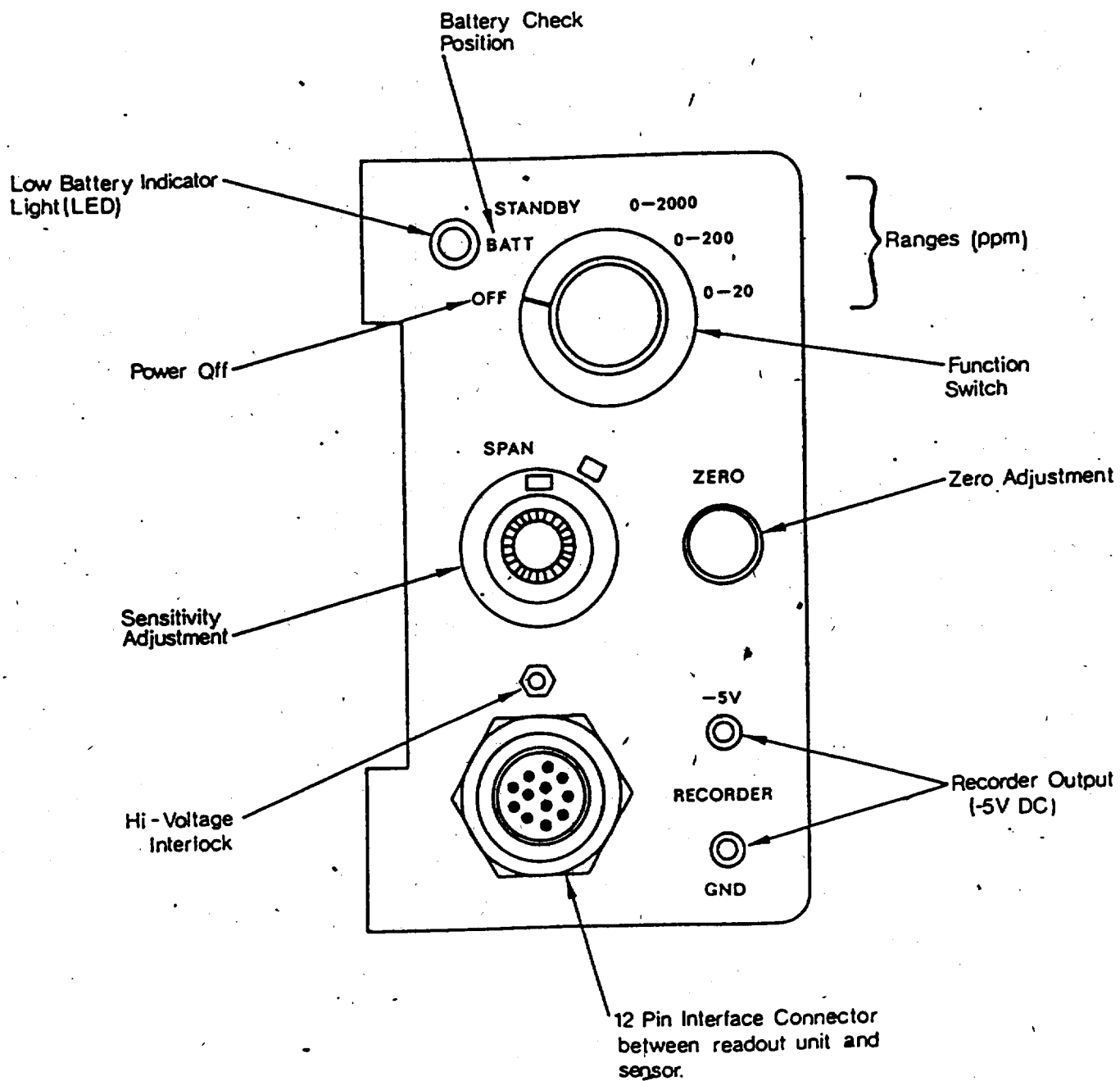
TABLE II

BRIEF DESCRIPTION OF INSTRUMENT
CONTROLS AND FUNCTIONS*

Control	Function
Six Position Switch	<p>OFF - Shuts off all power and removes DC voltages.</p> <p>ON - In any other function position or measuring mode, the electronics are on.</p> <p>BATTERY CHECK - Indicates the condition of the battery. If needle position is in lower portion of green battery arc, the instrument should be recharged.</p> <p>STANDBY - UV lamp is off but electronics are on. This position will conserve power and extend the useful operating time between recharges of the battery. This position is also utilized to adjust the electronic zero.</p> <p>RANGES - 0-20, 0-200, 0-2000 direct reading ranges available at minimum gain for benzene. More sensitivity is available by adjusting the span potentiometer.</p>
Zero Potentiometer	<p>A ten turn potentiometer is employed to adjust the zero electronically when the instrument is placed in the standby position with the probe attached. This eliminates the need for a hydrocarbon free gas.</p>
Span Potentiometer	<p>A ten turn counting potentiometer is utilized for upscale setting of the meter on calibration gas. Counter-clockwise rotation increases the sensitivity (~10 times). This pot can increase the sensitivity to make the instrument direct reading for nearly any gas which the instrument responds to.</p>

*For position of layout controls see Figure 2.

Figure 2 Control Panel Functions



To zero the instrument, turn the function switch to the standby position and rotate the zero potentiometer until the meter reads zero. Clockwise rotation of the zero potentiometer produces an upscale deflection while counterclockwise rotation yields a downscale deflection. Note: no zero gas is needed, since this is an electronic zero adjustment (see below). If the span adjustment setting is changed after the zero is set, the zero should be rechecked and adjusted, if necessary. Wait 15 or 20 seconds to ensure that the zero reading is stable. If necessary, readjust the zero.

The instrument is now ready for calibration or measurement by switching the function switch to the proper measurement range. The instrument is supplied calibrated to read directly in ppm (v/v) 0-20, 0-200, 0-2000 of benzene with the span position set at 9.8. For additional sensitivity, the span potentiometer is turned counterclockwise (smaller numbers) to increase the gain. By changing the span setting from 10.0 to 1.0 the sensitivity is increased approximately ten fold. Then, the 0-20, 0-200, and 0-2000 ppm scales become 0-2, 0-20, and 0-200 ppm full scale, respectively. This span control is also utilized to make the instrument scale read directly in ppm of the compound being measured. E.g., it is adjusted to match the value of a calibration gas to that same reading on the instrument scale. The span control can be utilized to calibrate nearly any compound, measured by photoionization, to be direct reading on the 0-20 ppm range. For example, gain settings of 4.5 or 8.9, respectively, will provide direct reading capability (0-20, 0-200 ppm) for vinyl chloride and trichloroethylene, respectively. For a listing of approximate gain setting values see Table IV.

A small DC operated fan is used to pull air through the photoionization sensor at a flow rate of three to seven hundred centimeters per minute (ca. 0.5 lpm). The fan provides nearly instantaneous response times (Figure 3) while consuming little power. The characteristics of a fan are such that it cannot tolerate a significant pressure drop without affecting the flow rate and therefore either the instrument reading or response time. Since photoionization is essentially a nondestructive technique, changes in flow rate do not affect the signal but if a large pressure drop is imposed at the inlet of the probe, the sample may not reach the sensor.

TABLE III

VERIFICATION OF ELECTRONIC ZERO FOR
PHOTOIONIZATION ANALYZER*

Sample	Instrument Reading (ppm)	% of F.S.
Room Air	0.7	35
Room Air Passed Through 6" x 3/4" OD Charcoal Scrubber	0.1	5
Zero Air	0.25	12.5
Zero Air Passed Through 6" x 3/4" OD Charcoal Scrubber	0.04	2

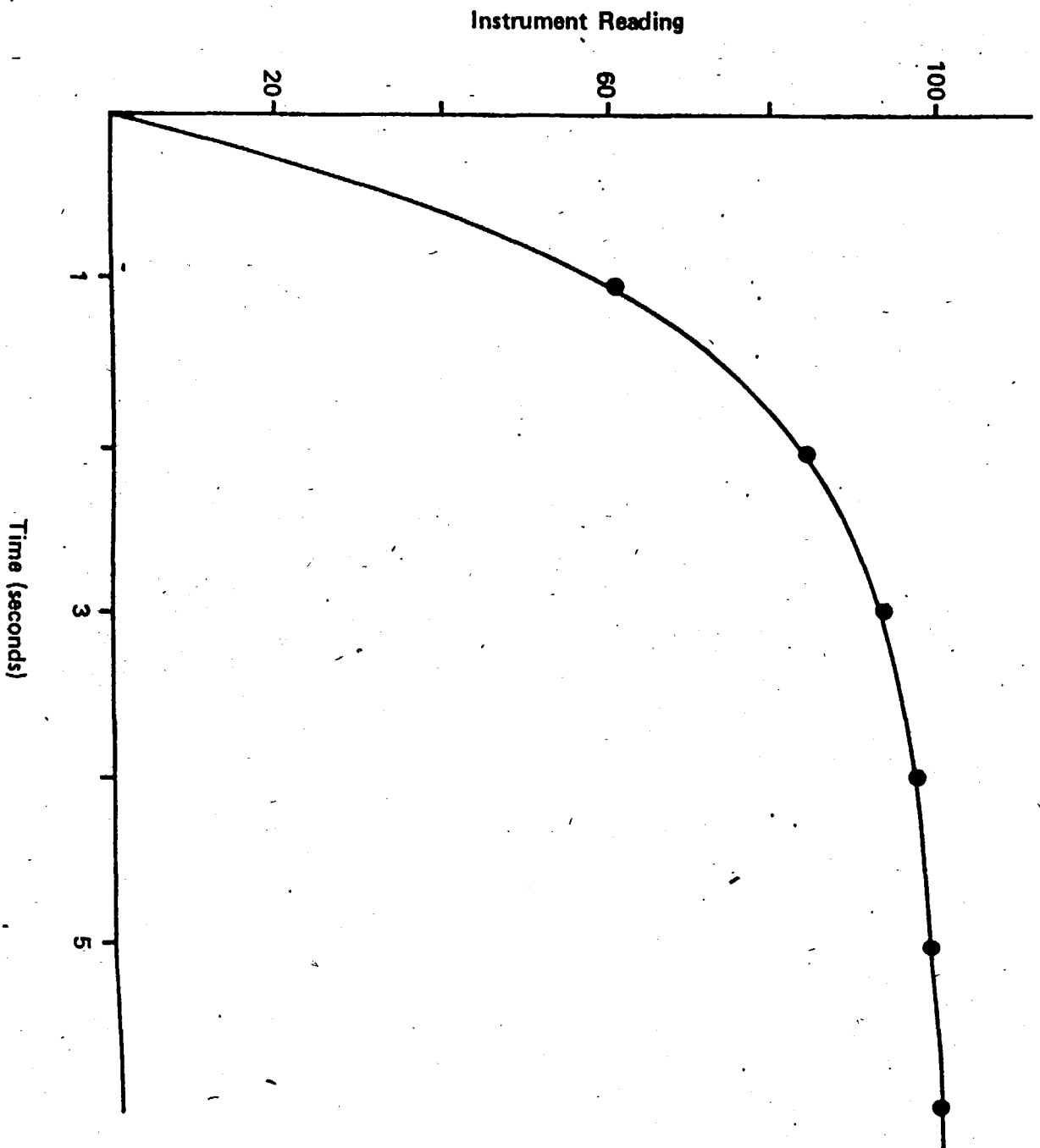
*Maximum Gain = 2 ppm full scale.

TABLE IV
RELATIVE PHOTOIONIZATION SENSITIVITIES*
FOR VARIOUS GASES

Chemical Grouping	Relative Sensitivity	Examples
Aromatic	10.0	Benzene, Toluene, Styrene
Aliphatic Amine	10.0	Diethylamine
Chlorinated Unsaturated	5-9	Vinyl Chloride, Vinylidene Chloride, Trichloroethylene
Carbonyl	5-7	MEK, MIBK, Acetone, Cyclohexene
Unsaturated	3-5	Acrolein, Propylene, Cyclohexene, Allyl Alcohol
Sulfide	3-5	Hydrogen Sulfide, Methyl Mercaptan
Paraffin (C ₅ -C ₇)	1-3	Pentane, Hexane, Heptane
Ammonia	0.3	—
Paraffin (C ₁ -C ₄)	0	Methane, Ethane . . .

*Sensitivities in ppm (v/v).

Figure 3. Time Response for the Photoionization Analyzer.



The instrument was designed to measure trace gases over a concentration range from less than 1 ppm to 2000 ppm. Higher levels of various gases (to percentage range) can be measured but the recommended procedure is to dilute the sample with clean air to a concentration of less than 500 ppm. This is generally within the linear range of the instrument, and if the measured concentration is multiplied by the dilution ratio the correct concentration in the stream can be determined. A typical calibration curve is shown in Figure 4. Note that the calibration curve for benzene (the photoionization standard) is linear (over more than three decades) up to about 600 ppm (v/v).

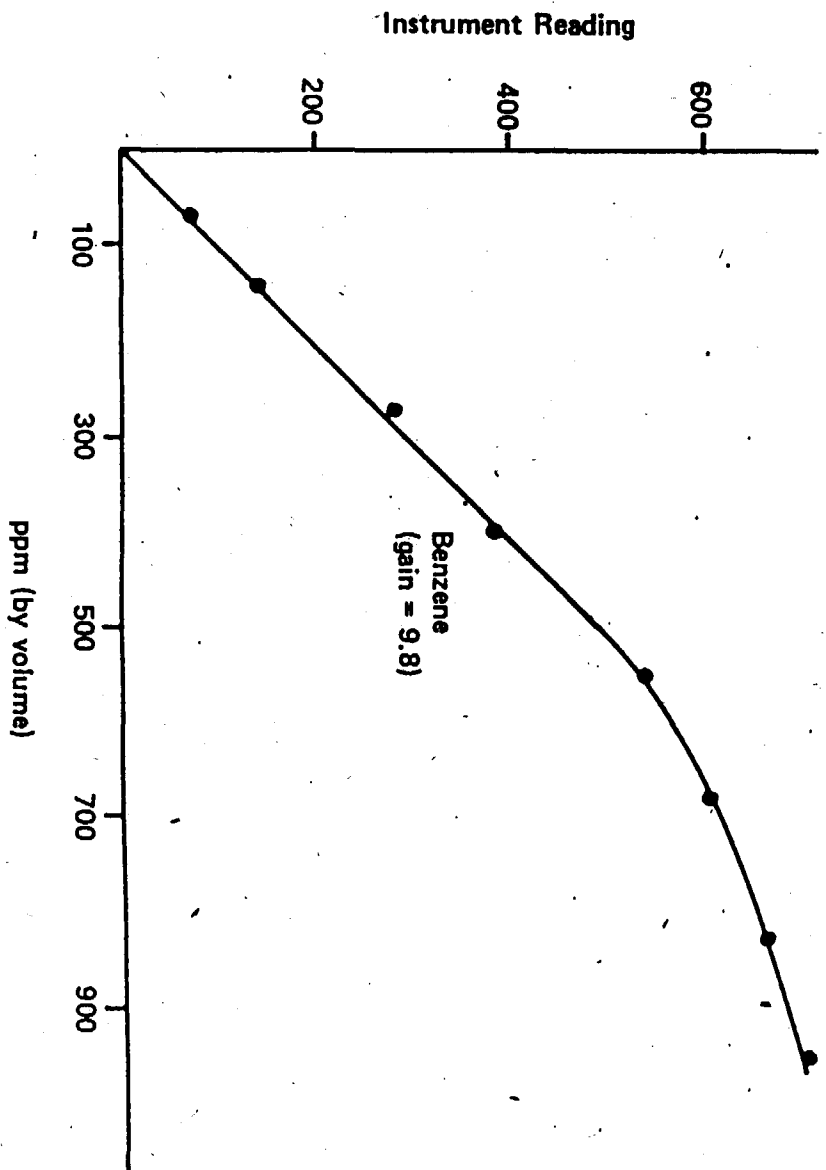
If the probe is held close to AC power lines or power transformers, an error may be observed. For measurements made in close proximity to such items, their effect on measurements can be determined by the following procedure. Zero the instrument in an electrically quiet area, in the standby position, then move the instrument to the questionable area involved. If AC pickup is going to be a problem, the meter (in the standby position) will indicate the magnitude of the error.

The instrument is equipped with an automatic solid state battery protection circuit. When the battery voltage drops below ~ 11 volts, this circuit will automatically turn off the power to the instrument. This prevents deep discharging of the battery and considerably extends the battery life. If the instrument is unintentionally left on overnight, the battery will be unharmed because of the battery protection circuit. If the instrument battery check reads low and the lamp doesn't fire, plug the charger into the instrument. The power to the analyzer should then be returned.

To charge the battery, place the mini phone plug into the jack on left side of the bezel prior to plugging charger into 120 VAC. When disconnecting charger, remove from 120 VAC before removing mini phone plug. The battery is completely recharged overnight (ca. 14 hours). To ensure that the charger is functioning, turn the function switch to the battery check position, place phone plug into jack and plug charger into AC outlet. The meter should go upscale if charger is working and is correctly inserted into the jack.

The instrument can be operated during the recharge cycle. This will lengthen the time required to completely recharge the instrument battery.

Figure 4. Typical Calibration Curve for Photoionization Analyzer.



SECTION 3

CALIBRATION

Static or dynamic gas generation systems can be utilized for calibration of the instrument. A number of such systems for generating test atmospheres for various gases have been described by G. O. Nelson in "Controlled Test Atmospheres," Ann Arbor Science Publishers, Ann Arbor, Michigan (1971).

The most convenient packages for calibration are the non-toxic analyzed gas mixtures available from HNU Systems in pressurized containers (Catalogue #101-350).

A rapid procedure for calibration involves bringing the probe and readout in close proximity to the calibration gas, cracking the valve on the tank and checking the instrument reading. This provides a useful spot check for the instrument.

The recommended and most accurate procedure for calibration of the instrument from a pressurized container is to connect one side of a "T" to the pressurized container of calibration gas, another side of the "T" to a rotameter and the third side of the "T" directly to the 8" extension to the photoionization probe (see Figure 5). Crack the valve of the pressurized container until a slight flow is indicated on the rotameter. The instrument draws in the volume of sample required for detection, and the flow in the rotameter indicates an excess of sample. Now adjust the span pot so that the instrument is reading the exact value of the calibration gas. (If the instrument span setting is changed, the instrument should be turned back to the standby position and the electronic zero should be readjusted, if necessary.)

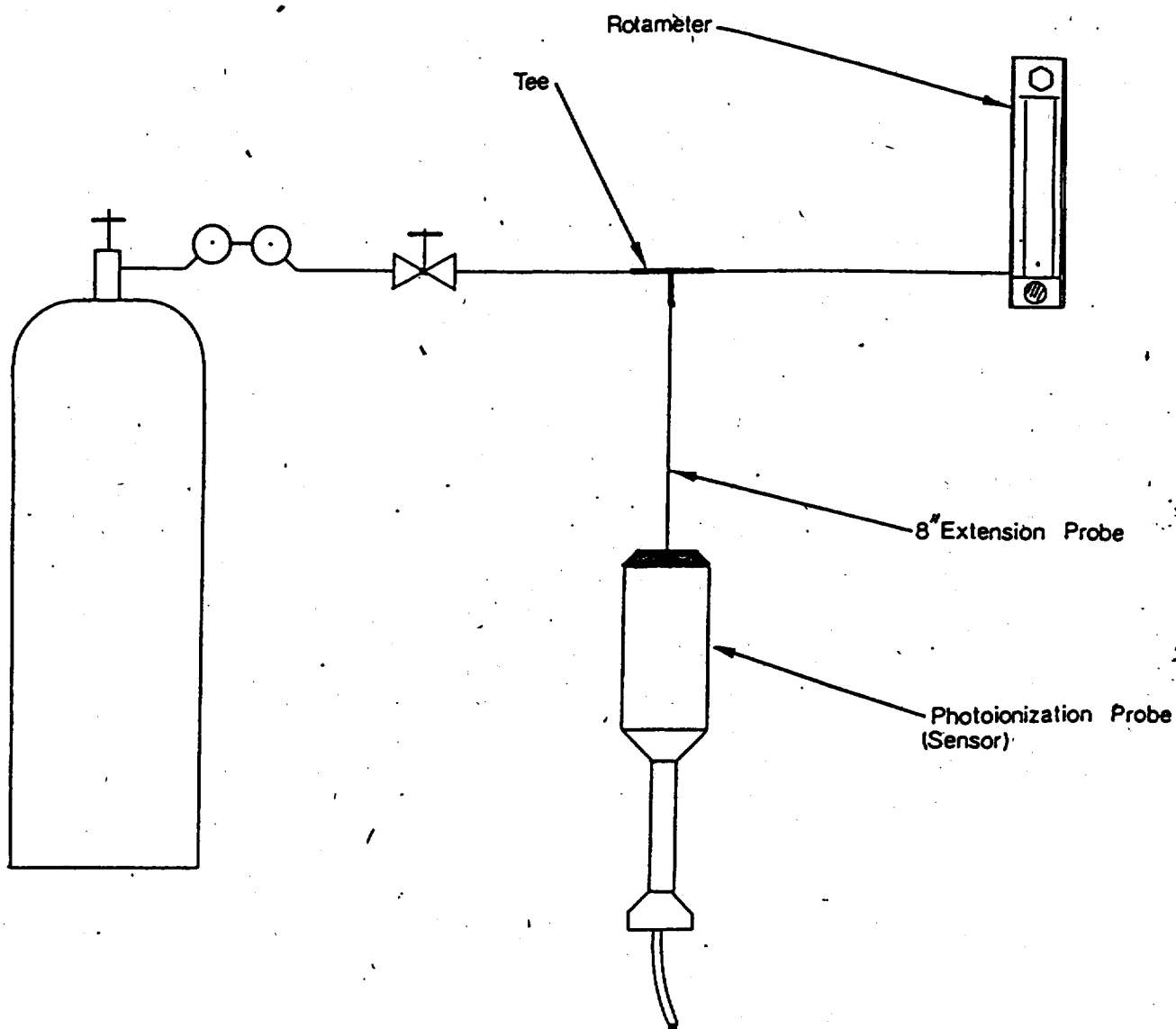


Figure 5. Recommended Calibration Procedure for Photoionization Analyzer

The calibration gas* should be prepared in the same matrix (air, nitrogen, hydrogen, etc.) in which it is to be measured, otherwise an inaccurate reading may be obtained. The increased response which is seen in oxygen free gases can be attributed to a reduction in the quenching of ions by oxygen (actually O_2^-) and is typical of any ionization detector. The quenching effect of oxygen is constant from about ten percent O_2 to very high levels.

If a gas standard prepared in nitrogen is to be used for measurements in air, fill a 0.5 or 1 liter bag with the standard then add 50 or 100 cc of pure oxygen to bring the level to 10-12%.

Any error between this value and 20% oxygen is quite small.

If the sample to be measured is in nitrogen, standards should be prepared in nitrogen. This will result in an increase in sensitivity of approximately 4.

* Calibration with toxic gases should be performed in a hood since the 101 is a non-destructive analyzer.

Appendix A-A5

Instruction Manual for the Mount Sopris Model 1000-C Borehole Geophysical Logging Device

INSTRUCTION MANUAL
MODEL 1000-C LOGGER

PORTABLE BOREHOLE LOGGER

MODEL 1000-C

Operation and Maintenance Manual

Serial Number 011 to Present

September 1977

MOUNT SOPRIS INSTRUMENT COMPANY

P. O. Box 449 Industrial Park
Delta, Colorado, U.S.A. 81416
Phone (303) 874-4852 TWX 910-929-6833

ADDENDUM

Changes for Model 1000-BP

1-10.0 Change the following items to read as shown below:

10.	120 vac Charging cable	1000-BP-120
10A.	240 vac Charging cable	1000-BP-240
11.	12 vdc Charging cable	A-500K 0107

2-6.1 Change paragraph to read as follows:

Connect the proper charging cable for the voltage source available to the BATTERY CHARGE connector (Fig. 1-10). If the 12 vdc cable is used, the red lead connects to the positive (+) terminal of the source and the black lead to the negative (-). Two charging cords are provided for charging from an a.c. source, one for 120 volts and the other for 240 volts. Select the proper cable and connect the free end as follows: Black to the a.c. HOT, white to the RETURN, and green to EARTH GROUND.

NOTE: If the polarity of the 12 vdc cable is reversed, no damage will result but the batteries will not be charged.

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SECTION I

GENERAL INFORMATION

1-1. INTRODUCTION

The Model 1000-C is a complete, fully portable, lightweight (27.3 Kg-60 lbs) backpack mountable, borehole logging unit.

Using the standard probe (G375/A), it is possible to record up to three different logs in one trip in the hole: (1) Gross count, dead-time corrected gamma radiation, (2) Self Potential, and (3) Single Point Resistance. Other Nuclear Pulse counting tools available include gamma-gamma and neutron-neutron.

The recorder is a dual pen, servo-driven type, equipped with a bi-directional chart drive allowing multiple logs and re-runs without resetting the chart paper.

The power requirements for the Model 1000-C are provided by internal nickel cadmium batteries which are recharged by a built-in battery charger when connected to an A.C. or D.C. source. Battery life will provide a minimum of eight (8) hours of logging.

The hand cranked winch has two speeds: a 1:1 ratio for going down hole, and a 4:1 ratio for coming back out. The winch comes with 305 M (1000 ft.) of steel armored logging cable.

The Model 1000-C, when handled reasonably and maintained properly, will provide many years of reliable logging.

1-2.0 GENERAL SPECIFICATIONS

The Model 1000-C logger comes equipped with one combination probe, 305 M (1000') of cable, recorder-instrument assembly, shipping containers, spare parts and consumables, and all necessary cables and hardware required to log a borehole. A backpack frame with necessary fittings is optionally available.

1-2.1 Shipping Weight: 36.4 Kg. (80 lbs.)

1-2.2 Net Weight (With 305 M of cable and backpack frame): 27.3 kg. (60 lbs.)

1-2.3 Shipping Dimensions: Probe/Accessory Case = 33cm x 124.5cm x 10.2cm (13" x 49" x 4")

Winch/Recorder Case = 58.4cm x 48.3cm x 43.2cm (23" x 19" x 17")

1-2.4 Operating Temperature Range (limited by pens): -25°C (-13°F) to 55°C (131°F)

1-3.0 WINCH

1-3.1 Cable Capacity: 305 M (1000')

1-3.2 Cable Type: U. S. Steel type no. 1N10SB (or equivalent) galvanized steel armored logging cable. Single center conductor, D. C. resistance approximately 24Ω/305M. Tensile strength is 408 Kg. (900 lbs.).

1-3.3 Cable Head: Tapered cone and sleeve strain relief. Cable center conductor is water sealed with a Mecca underwater connector. Connection to probe is through center contact and outer screw threads (3/4 - 16). Outside diameter is 2.86 cm (1.125").

1-3.4 Hand Crank Gearing: Direct drive (1:1 ratio) or step down (4:1 ratio) selected with easily removable crank.

1-4.0 RECORDER INSTRUMENT ASSEMBLY

1-4.1 Depth Odometer: 5 digit bi-directional, resettable, mechanical odometer geared directly to the measuring wheel. Resolution is 0.1 M (0.1' on English models).

1-4.2 Measuring System: Hard anodized grooved aluminum wheel. The measuring wheel rotates 3 1/3 revolutions per meter (3' on English models).

1-4.3 Servo Amplifier (Recorder): Two pen, non-overlapping, servo-driven, rectilinear recorder.

1-4.4 Recorder Response Time: Time to full scale (step function in) is ~1 second.

1-4.5 Pens: Disposable felt tip cartridge available in red and black.

Section I (1000-C)

- 1-4.6 Pen lifter: Both pens operate simultaneously with front panel lever.
- 1-4.7 Chart Drive: Bi-directional, geared (through multi-tooth clutch) to measuring wheel. Turned on, and off with front panel shift lever.
- 1-4.8 Chart Paper: 10 cm wide grid Z-fold paper with 1 cm x 1 cm major divisions and 0.5 cm intermediate lines. Each box contains two packets (7.6 m - 25' each) of paper. The last 61 cm (2') are marked with a red line on the right-hand margin.
- 1-4.9 Sensitivity: 100 mV. (F. S.)

1-5.0 GAMMA CIRCUIT

- 1-5.1 Ranges: 5 cps/div. (50 cps full scale) to 5 Kcps/div. (50 Kcps full scale) in 1-2-5 ratio steps.
- 1-5.2 Internal Calibration Source: 5.000Khz. square wave crystal oscillator with a temperature coefficient of $\pm 0.01\%/^{\circ}\text{C}$ maximum.
- 1-5.3 Temperature Coefficient (total circuit): Each unit individually aligned for $\pm 0.02\%/^{\circ}\text{C}$ maximum from -25°C to $+75^{\circ}\text{C}$.
- 1-5.4 Time Constant: 2 pole Bessel function active filter. Full scale response time is ~ 7 seconds (step function in).
- 1-5.5 Deadtime Correction: Realtime analog correction adjusted to < 1 microsecond as calculated by the Energy Research and Development Administration in their test pits at Grand Junction, Colorado.
- 1-5.6 Input Sensitivity: ± 380 mV ± 10 mV.
- 1-5.7 Downhole Power: 30 vdc ± 1 v at 50 ma. maximum as measured at the slip rings (with combination probe connected).

1-6.0 SELF POTENTIAL

- 1-6.1 Ranges: 2mV./div. (20mV full scale) to 100 mV/div. (1v. full scale) in 1-2-5 ratio steps.
- 1-6.2 Bias Range: 0 to 500 mV + or - as selected with POLARITY switch.
- 1-6.3 Time Constant: 2 pole Bessel function active filter. Full scale response time is ~ 1 second (step function in).
- 1-6.4 Input Impedance: > 2 megohms.

1-7.0 SINGLE POINT RESISTANCE

- 1-7.1 Range: $2\Omega/\text{div.}$ (20 ohms full scale) to $100\Omega/\text{div.}$ (1000 ohms full scale) in 1-2-5 ratio steps.
- 1-7.2 Displacement: 0 to 500 ohms maximum.
- 1-7.3 Time Constant: 2 pole Bessel function active filter. Full scale response time is ~ 1 second (step function in).
- 1-7.4 Current Generator Output: Constant current (varies with range switch setting--20mA max.) at 25 hz to 30 hz (selected by internal switch).
- 1-7.5 Resistance Voltmeter Input Impedance: >500 K ohms.

1-8.0 BATTERIES & BATTERY CHARGER

- 1-8.1 Battery Type: General Electric type GCW3.5SB (or equivalent) Nickel Cadmium batteries (10 each).
- 1-8.2 Battery Rating: 1.2vdc at 3.5 Ah.
- 1-8.3 Battery Life: 8 hours continuous operation minimum. Varies with log(s) being run. Maximum drain is during SP-R log.
- 1-8.4 Battery Charger Input: 12 to 14 vdc at 1.0 A maximum, 120 vac at 0.10 A max., and 220 vac at 0.05 A maximum (A. C. input is 50 to 400 hz.).
- 1-8.5 Battery Charger Output: Dual, constant current outputs. 350 mA each.
- 1-8.6 Charging time to full charge: 12 to 14 hours.

1-9.0 PROBES

1-9.1 G375/A Standard Combination Probe

- 1-9.1.1 Dimensions: 1.1 m (43.5") long by 3.18 cm (1.25") maximum outside diameter including neoprene insulating sheath. The stainless steel housing is 2.86 cm (1.125") diameter.
- 1-9.1.2 Weight: 2.95 Kg (6.5 lbs.).
- 1-9.1.3 Construction: Stainless steel (type 303) housing with lead electrode for SP and R logs. O-ring sealed at all joints.
- 1-9.1.4 Power Requirement: 20 vdc (at cable head) minimum at 35 mA. Maximum voltage in is 30 vdc, 24 volts is nominal.
- 1-9.1.5 Scintillation Crystal: 38.1mm (1.50") long by 12.7mm (0.5") diameter sodium iodide, thallium activated scintillation crystal in ruggedized, sealed, aluminum mount.

Section 1 (1000-C)

1-9.1.6 Electrical Connection: Insulated center spring loaded contact and mechanical threaded attachment to cable head. Center contact is positive.

1-9.1.7 Output: 6.5v 1 microsecond wide positive pulse superimposed on the positive supply line (center conductor).

1-9.1.8 Deadtime: Constant 5.0 microseconds.

1-9.1.9 K-factor: Average K-factor at 10 cm intervals is 4.8×10^{-6} (2.5×10^{-5} at 0.5' intervals).

1-9.2 G375/A-1.0 1" O.D. Combination-Probe

1-9.2.1 Dimensions: 1.1m (43.5") long by 2.54cm (1.0") outside diameter stainless steel housing (for very small diameter holes). The probe must be covered with a neoprene sheath or electrical tape to run the SP and R logs.

1-9.2.2 Weight 2.7 Kg (6 lbs.).

1-9.2.3 K-factor: Average K-factor at 10 cm intervals is 5.2×10^{-6} (2.6×10^{-5} at 0.5' intervals).

All other characteristics are the same as Model G375/A.

1-9.3 G375/AS Stratigraphic Combination Probe

1-9.3.1 Dimensions: 1.02m (40.2") long by 41.1 cm (1.62") maximum outside diameter including neoprene sheath (housing is 38.1cm (1.50") diameter.

1-9.3.2 Weight: 4.4 Kg (9.7 lbs.).

1-9.3.3 Scintillation Crystal: 76.2mm (3.0") long by 22.2 mm (0.875") diameter sodium iodide, thallium activated scintillation crystal in ruggedized, sealed aluminum mount.

All other characteristics are the same as Model G375/A.

1-10.0 CONSUMABLES AND SPARE PARTS

Item	Description	Part No.	Quantity*
1.	Chart Paper	BP-10	3 Boxes
2.	Pen Cartridge-Black (Center Nib, long)	120-430	5 Each
3.	Pen Cartridge-Black (Nib on Right, long)	130-430	5 Each
4.	Pen Cartridge-Red (Offset Right, long)	130-430	5 Each
5.	Adjustment Screwdriver	R-3324	1 Each
6.	Rubber Cable Wiper Balls	RB-1.875	2 Each
7.	Surface Electrode Assembly	B-500K 0125	1 Each
8.	Mecca Sockets	2670-6	3 Each
9.	Mecca Boot	2458-1	1 Each
10.	A. C. Charging Cable	A-500K 0124	1 Each
11.	12vdc Charging Cable	B-500K 0108	1 Each
12.	Cablehead Protector Cap	B-2000 0108	1 Each
13.	Probe Top Protector Plug	B-2000 0107	1 Each
14.	Hand Crank for Winch		1 Each
15.	O-Ring (for cable head)	2-115	1 Each
16.	Nylon Accessory Storage Bag		1 Each
17.	Recorder Protective Cover		1 Each
18.	Operation and Maintenance Manual (1000-C)		1 Each
19.	Winch-Recorder Shipping Case		1 Each
20.	Probe-Accessory Shipping Case		1 Each
21.	Standard Combination Probe	G375/A	1 Each
22.	Backpack Frame		Optional
23.	1" O.D. Combination Probe	G375A-1.0	Optional
24.	Stratigraphic Combination Probe	G375/AS	Optional
25.	Filtered Combination Probe	G375F/A	Optional

When logging in extremely remote areas, you may wish to consult Mount Sopris for a list of spare parts (including both electronic and mechanical components to allow repairs to be made in the field.

*NOTE: This quantity refers to the number of pieces shipped with each unit initially.

SECTION II

OPERATING INSTRUCTIONS

2-1.0 Incoming Inspection

The Model 1000-C should be unpacked and inspected as soon as possible. Check externally for broken knobs, bent shafts or levers, damaged connectors, etc. Also check for scratches, dents, or any gross misalignment of cases, shafts, plates, etc.

A quick incoming test may be performed as follows:

2-1.1 Chart Drive and Depth Measuring System

Shift the CHART DRIVE lever (fig. 1-13) to ON. CAUTION: do not force the lever. If necessary, rotate the CHART ADVANCE wheel (fig. 1-22) slightly while pushing the CHART DRIVE lever to ON. Reset the DEPTH odometer (fig. 1-12) to zero with the RESET wheel (fig. 1-11). Note the position of one of the pens on the chart, and rotate the MEASURING WHEEL (fig. 1-23) counterclockwise exactly 10 revolutions. The chart will move 3 cm and the DEPTH odometer will read 9997.0 meters (9991.C' on English models). Rotating the MEASURING WHEEL clockwise exactly 10 revolutions brings the chart and DEPTH odometer back to zero.

2-1.2 Recorder-Instrument Circuitry

Connect one of the combination probes to the cable head as shown in figure 4. Connect the surface electrode to the front panel connector (fig. 1-10) and short this electrode to the electrode on the probe (fig. 1-4). Turn the CPS/DIV. switch (fig. 1-1) to CAL, gamma DISPLACEMENT switch (fig. 1-3) to zero, and the LOG SELECTOR (fig. 1-9) to γ . The right-hand pen will go completely off scale to the right, and the left pen will slowly go to full scale (27 seconds). Depress the left ZERO button (fig. 1-16) and see that the left ZERO control (fig. 1-15) adjusts the pen to the left and right of zero (left-hand margin). Set the left pen to zero. Turn the CPS/Div. switch to 10 (100 cps full scale). The recorder will indicate the level of background radiation. Turn the LOG SELECTOR to POWER OFF and set the following controls as indicated: POLARITY switch (fig. 1-5) to "+", BIAS control (fig. 1-6) to zero, MV/DIV. switch (fig. 1-4) to 10, Ω /DIV. switch (fig. 1-7) to 10, and resistance DISPLACEMENT control (fig. 1-8) to zero.

Turn the LOG SELECTOR to SP-R. The left pen (SP) will read near the left margin, and the right pen (R) reads up scale about 8 cm. If the right pen goes off scale to the right, check the connection between the surface electrode and probe electrode. Depress the right ZERO button (fig. 1-19) and see that the right ZERO control (fig. 1-18) will adjust the pen both left and right of the center of the chart. The zero line for the right pen is the center of the chart. Set the right pen on "zero". Turning the BIAS control clockwise will cause the SP pen to go up scale (to the right) on "+" POLARITY and down scale on "-" POLARITY.

Turning the resistance DISPLACEMENT control clockwise causes the R pen to go down scale.

Should any of the aforementioned tests fail, check to see if the LOW BATTERY indicator (fig. 1-14) is on (LOG SELECTOR on γ or SP-R); if it is, recharge the batteries (refer to sec. 1-8). Otherwise, refer to section 3-9. It is normal for the LOW BATTERY indicator (fig. 1-14) to flash as the LOG SELECTOR is switched from POWER OFF to γ or SP-R.

2-1.3 Storage

The logger should be stored in a temperate, dry area if possible. The temperature range for storage is -40°C (-40°F) to 70°C (158°F). All protective covers should be in place and the units stored in their shipping containers. The batteries should be given a full charge (12 to 14 hrs.) prior to storage and re-charged (at least 8 hours) every six months.

2-2.0 OPERATING CONTROLS

Refer to Fig. 1	Control	Function
1.	CPS/DIV:	Gamma range switch, selects the number of counts per second per division (1cm) to be recorded on the chart. In the CAL. position, the internal calibration oscillator is connected to the input of the gamma circuitry and causes the pen to go full scale (right-hand margin).
2.	Gamma CAL.:	20 turn screwdriver adjustment to set the gamma pen on full scale with the CPS/DIV. switch in the CAL. position.
3.	Gamma DISPLACEMENT:	Displaces the gamma pen down scale (to the left). The amount of displacement is equal to the CPS/DIV. switch setting (100/div. maximum) multiplied by the gamma DISPLACEMENT switch setting i.e., if the CPS/DIV. switch is on 20 and the DISPLACEMENT switch is on 15, the pen is displaced 300 cps ($1\frac{1}{2}$ times full scale) to the left. CAUTION: This switch must be kept on zero when the CPS/DIV. switch is on 200 or above.
4.	MV/DIV.:	Self Potential range switch. Selects the number of millivolts per division (1cm) to be recorded. Full scale for the SP pen is assumed to be at the center of the chart.
5.	BIAS POLARITY:	Selects the polarity of the bias signal fed to the SP circuit.
6.	BIAS:	Controls the amount of bias signal fed to the SP circuit. The BIAS range is 0 to 500 mv.
7.	/DIV.:	Resistance range switch. Selects the number of ohms per division (1cm) to be recorded. Zero for the R pen is assumed to be the center of the chart; full scale is the right-hand margin.
8.	Resistance DISPLACEMENT:	Controls the number of ohms and R pen is displaced to the left. The range is 0 to 500 ohms.

Refer to Fig. 1	Control	Function
9.	LOG SELECTOR:	Controls the power to all circuitry and selects gamma or SP-R operation. This switch must be in POWER OFF to allow charging.
10.	BATTERY CHARGE-SURFACE ELECTRODE:	A connector which provides connections for all battery charging and the surface electrode.
11.	RESET:	Allows resetting DEPTH odometer to zero.
12.	DEPTH:	Displays the amount of cable which has been spooled off the winch drum from the time the odometer was reset.
13.	CHART DRIVE:	Controls multi-tooth clutch to engage or disengage chart. The chart will move at a ratio of 1 meter of borehole to 1 centimeter of chart. CAUTION: Do not force the CHART DRIVE lever to ON. If necessary, rotate the CHART ADVANCE wheel slightly (to line up a multi-tooth clutch) while pushing the lever to ON.
14.	LOW BATTERY:	Indicator will come on when batteries are low. A maximum of one hour of use remains after the indicator comes on.
15 & 18.	ZERO control:	Adjusts associated pen to zero. Zero for the left pen is the left-hand margin; zero for the right pen is the center of the chart.
16. & 19.	ZERO button:	When depressed, it shorts the input of the associated servo amplifier to ground (causing the pen to read zero).
17. & 20.	SERVO GAIN:	Adjusts the amount of gain in the feedback loop of the servo amplifier.
21.	PEN LIFTER:	Mechanically raises and lowers both pens.
22.	CHART ADVANCE:	Thumbwheel to manually move the chart. CAUTION: The CHART DRIVE must be OFF.

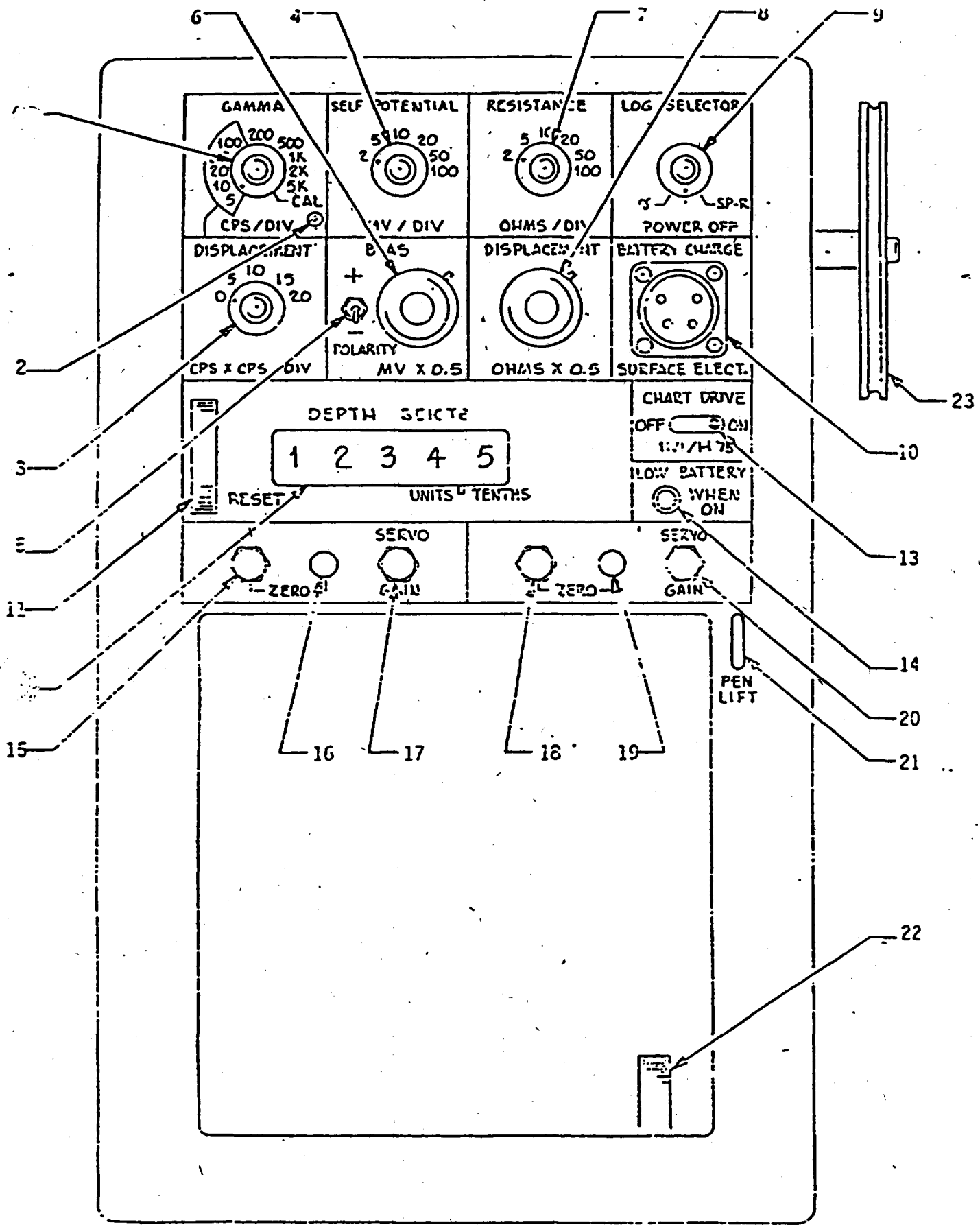


Fig. 1

Recorder Front Panel

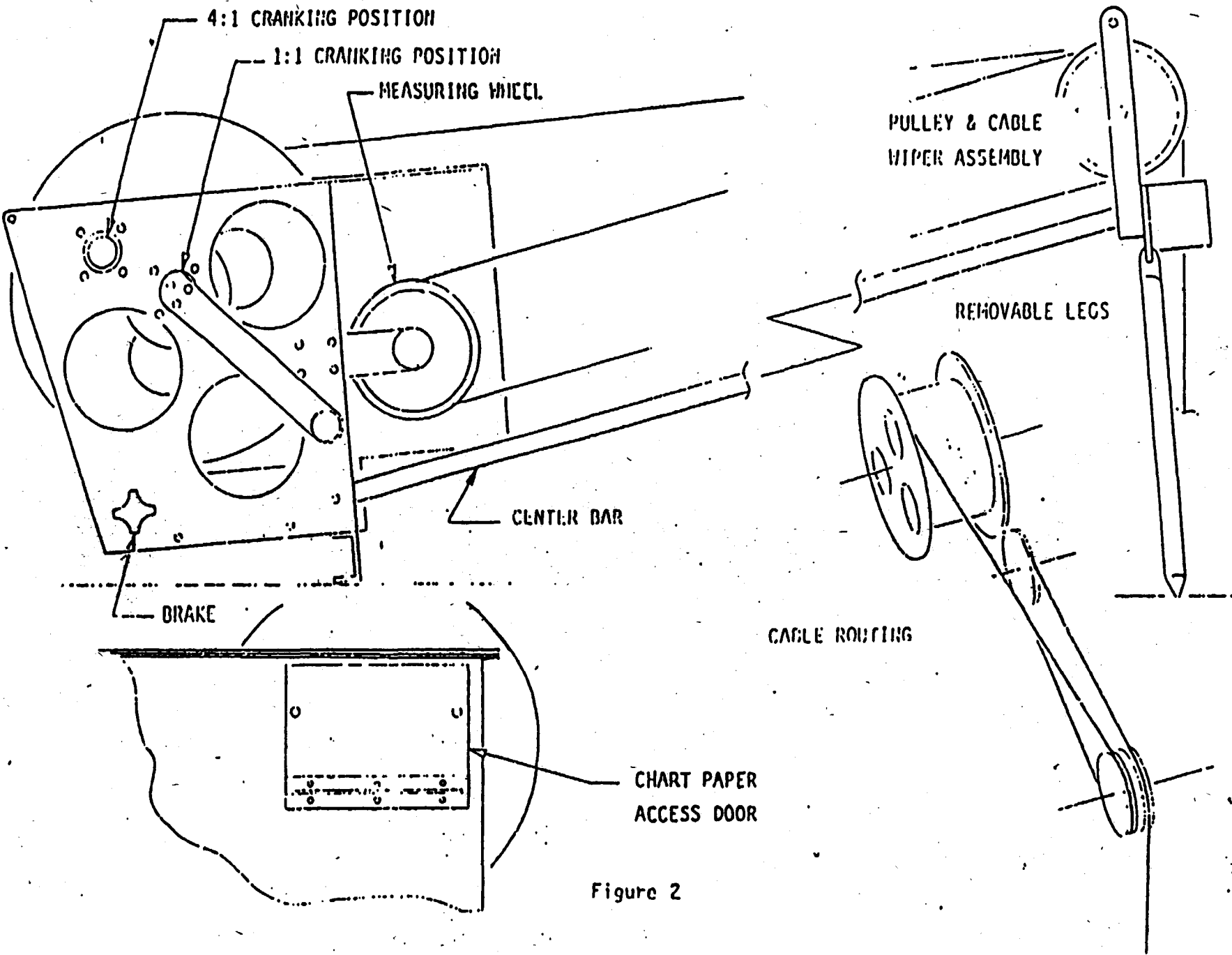


Figure 2

2-3.0 Replacing Chart Paper & Pens

2-3.1 Loading Chart Paper (refer to figure 3)

It may be easier, but not necessary, to remove chart pens before loading recorder with new chart paper.

Shift the CHART DRIVE lever (refer to figure 1) to "OFF" and open the side door. Rotate chart paper thumb wheel clockwise while pushing up, from the inside, on the front end of the Teflon paper holder. The rotating sprockets will catch the paper holder and lift it up and off of the front sprocket roller. The paper holder may rub along the left side of the opening in the front panel.

The bottom end of the paper holder remains inside the recorder and around the rear sprocket roller. Do not attempt to completely remove paper holder from recorder.

Load a new packet of chart paper into recorder and thread paper up and over the front roller. Rotate thumb wheel clockwise and feed the chart paper until it engages the rear roller. Be sure the paper is flat and properly aligned on sprockets before lowering the front end of the paper holder. Maneuver paper holder down over the front roller until it catches on sprockets. The thumb wheel may then be rotated in a counter-clockwise direction. It may be necessary to hold the pens up to clear the paper holder. The paper holder will then snap into place.

Use thumb wheel to feed chart paper through recorder and into take-up magazine. Make note of which way chart paper unfolds, so that you can get paper refolding into original position.

CAUTION:

Take care not to bend the paper holder. The stretching required for the paper holder to snap on and off of roller is normal. When the paper holder is in its normal operating position, it should fit slightly loose. If the paper wrinkles or tears, or if the paper holder has a tendency to lift up during operation, the paper holder has become bent, and must be resored to its original shape.

2-3.3 Pens (Refer to Fig.1)

Turn LOG SELECTOR to POWER OFF. The pens can then be removed by grasping the front of the pen cartridge and pulling straight out. Replace with a fresh cartridge in the reverse order. When not in use, the protective caps should be kept on the pen tips to prevent the pens from drying out.

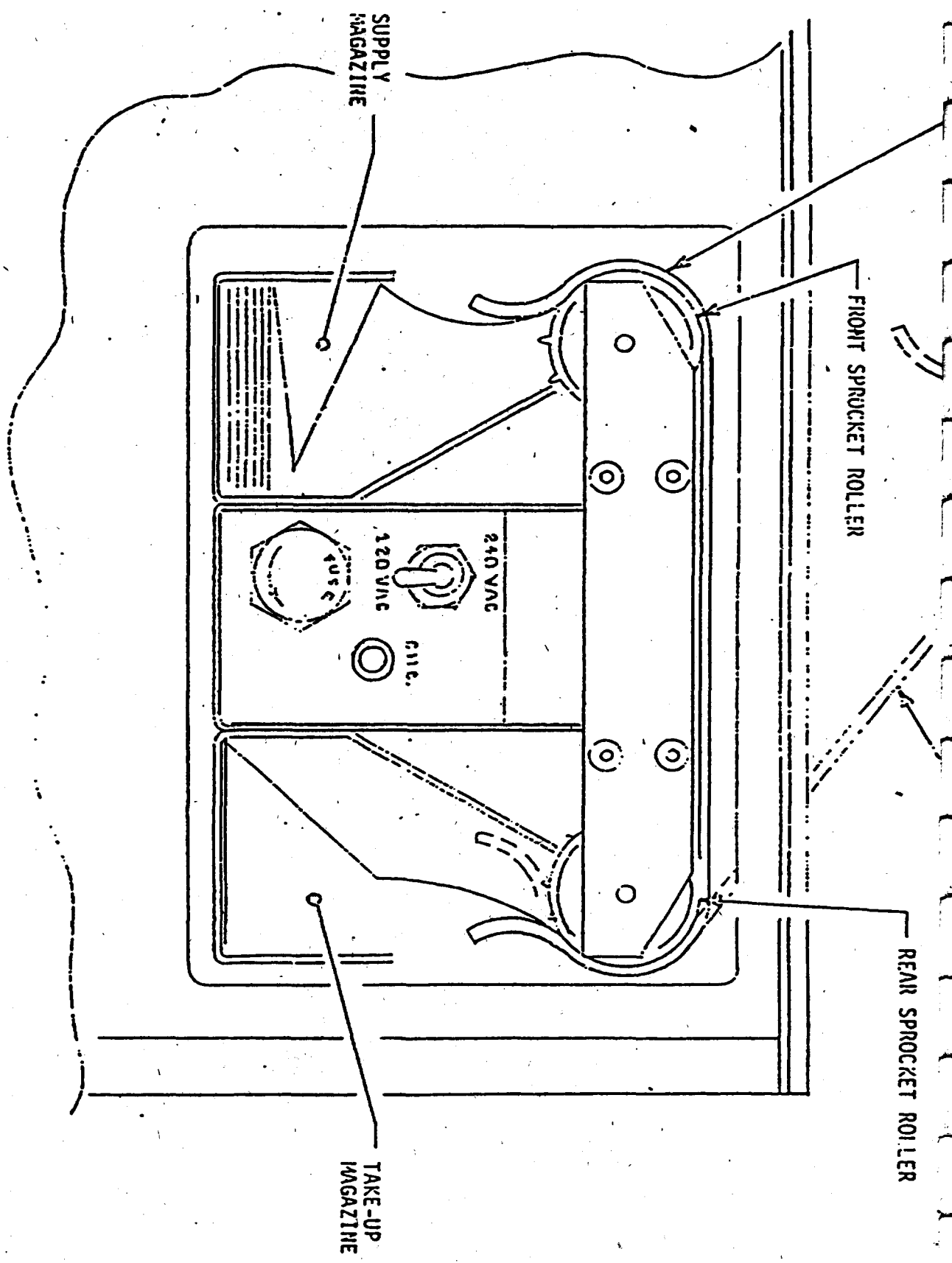


Fig. 3 CHART TRANSPORT ASSEMBLY (A)

2-4.0 Logging a Borehole

INTRODUCTION

- 2-4.1 The main objective for the operator of a logging unit is to produce an accurate, informative, and easy-to-read log. This means the pens make maximum use of the available chart space (full chart for Gamma and half chart each for SP and R), and do not overlap or criss-cross so much that they confuse the log. To do this requires selecting proper scales and setting bias or displacement controls properly (there is no substitute for experience). Using black pens for SP and R and a red pen for Gamma also helps keep the record straight. Since the two pens are non-overlapping, they may collide while logging SP and R. It is almost impossible to prevent this, so (if they do not separate naturally within a meter or so) the pens must be separated, using the SP BIAS control and/or R DISPLACEMENT control. Turning the R DISPLACEMENT control clockwise will cause the R pen to move to the left. Turning the SP BIAS control clockwise, with the POLARITY switch on "+", will move the SP pen to the right. With the POLARITY switch on "-" the pen will move to the left when the SP BIAS control is turned clockwise. A sample of a good log is illustrated in figure 5A and a poor log in 5B.

All three logs (Gamma, SP and R) can be run in one trip in the borehole by logging SP and R down, and Gamma on the way out. However, because of the added difficulty with this method (the hole cannot be pre-viewed on the way down to set up and SP and R controls, extra chart paper must be fed into the take-up magazine because the paper will run backwards while going down hole, etc.) it is recommended only when logging in poorly consolidated material, or when time is the ruling factor.

Prior to going to the drill site, check to make sure you have: fully charged batteries, a charging cable, spare pens (in both styles and colors), the winch crank, probe(s), extra chart paper, cable wiper balls, mud electrode, Gamma calibration screwdriver, electrical tape, and silicone grease (for O-Ring on cable head).

The following list is intended as a guideline to log a typical borehole, using the two trip method. (The probe is lowered to the bottom of the hole and Gamma is logged while coming out; the probe is then lowered a second time to log SP and R.

- 2-4.2 Clear the area around the borehole to give you a relatively clean, dry place to work.
- 2-4.3 Release the BRAKE and unspool a couple of meters of cable to give you enough slack to connect the probe and set up the tripod as shown in figure 2.
- 2-4.4 Place the logger on the borehole. Depending on the site, you may have to use scrap lumber, rocks, etc. to make the unit as level as possible.

- 2-4.5 Shift the CHART DRIVE lever to OFF. Check to see if there is enough paper in the supply magazine to log the hole.
- 2-4.6 Remove the protector plug from the probe and check to see that the threads and area around the contact are clean. Remove the knurled cable head protector (clean the threads and contact if dirty), apply a generous amount of silicone grease to the O-ring, and screw the probe to the cable head HAND TIGHT ONLY--do not use wrenches. The probe must be insulated with electrical tape as shown in figure 4.
- 2-4.7 Bury the mud plug by digging a small hole, putting the mud plug in, and filling the hole with dirt and fresh water. The mud pit will provide a good ground on freshly drilled holes. It may be necessary to use salt water to obtain a good ground in extremely arid areas. This must be noted on the log heading, as it usually reverses the polarity of the SP log.
- 2-4.8 Load the chart paper (refer to section II, paragraph 3) and put your log heading on it. A typical log heading might contain the unit serial number, probe serial number, probe K factor, type of logs run and their scales, hole number and location, operator's name, and the date.
- 2-4.9 The first log to run is Gamma, because it is usually considered most important, and depending on conditions in the borehole, one run may be all you can get. For clarity, the Gamma should be run with a red pen (SP and R in black). Remove the protective cap from the gamma pen and switch the LOG SELECTOR to γ . The right-hand pen will automatically go off scale to the right to allow full use of the chart for gamma.
- 2-4.10 The SERVO GAIN control (one for each channel) should be set as high as possible (clockwise) without having the recorder pens oscillate. If a recorder pen starts to oscillate, reduce the gain (counterclockwise) just to the point where the oscillation stops.
- 2-4.11 To check the gamma calibration, turn the CPS/DIV. switch to CAL. and alternately check for zero on the left-hand margin and full scale on the right-hand margin. Depress the left-hand ZERO button and adjust the ZERO control so the pen rests on the left-hand margin. Release the button, and the pen will come to rest near the right-hand margin. Adjust the pen for full scale with the CAL. screwdriver adjustment. Depress the ZERO button and again check for zero and then full scale to insure accurate calibration. The calibration procedure routinely should be checked before each hole.
- 2-4.12 Make certain the brake is set, and lower the probe into the borehole. Position the logger as necessary to center the cable in the hole. Slip the winch crank on the 1:1 ratio shaft, release the brake and crank the top of the cable head back up to ground level. Set the brake.

- 2-4.13 Reset the DEPTH odometer by rotating the depth RESET wheel upward, and then set in the distance from the top of the cable head, (ground level) to the reference point by manually rotating the measuring wheel. The gamma log and SP-R logs will be displaced by the distance between the center of the scintillation crystal to the center of the electrode (See Fig. 4). If this distance is considered significant, it may be compensated for by sliding the pens in or out slightly as the case may be. A good range for γ exploration is 20 cps/DIV.
- 2-4.14 You are now ready to lower the probe to the bottom of the hole. CAUTION: do not allow the probe to freewheel down the hole. Holding the winch crank, release the brake and crank the probe to the bottom of the hole. The bottom will be detected by the sudden loss of weight. The approximate depth can be obtained from the drilling crew.
- 2-4.15 When you hit bottom, take the slack out of the cable and set the brake. Move the winch crank to the 4:1 ratio shaft.
- 2-4.16 Adjust the chart with the CHART ADVANCE wheel so the pen will cross a major division on the chart (every 1 cm) at the same time the depth odometer indicates a whole number of meters (or at 3' intervals on English models). This makes reading the chart easier because each centimeter line will be an even depth reading i.e., every meter 273.0, 272.0, 271.0, (or 273.0, 270.0, --- 6.0, 3.0, 0.0 on English models). Shift the CHART DRIVE to ON. CAUTION: Do not force the CHART DRIVE lever. If necessary, rotate the CHART ADVANCE wheel very slightly (to line up a multi-tooth clutch) while shifting the CHART DRIVE lever to ON.
- 2-4.17 Check to be sure a good cable wiper ball is in place. If not, rotate the old one (or install a new ball) and cut a slice for the cable to go through.
- 2-4.18 You are now ready to make the first log (gamma) in the hole. Lower the pen, release the brake, and begin to crank the probe back out. A good speed for general logging is 4M/min. (15'/min.). This can be approximated by making one revolution of the crank every 2 seconds. As you come out of the hole, an attempt should be made to keep the cable as neat and level across the winch drum as possible.
- 2-4.19 If a gamma anomaly is encountered, the gamma pen will go off scale to the right. Continue to log until the pen comes back on scale and is reading the normal background gain. Stop cranking, raise the pen, and, while observing the pen, crank back down through the anomaly, selecting a scale which will keep the pen on scale in the upper half of the chart. Make a note of this range setting on the chart. When the pen is reading background (below the anomaly on the new scale), put the pen down and crank back up through the anomaly until the pen again returns to background. A re-run should be logged at 1 to 2 m/min (3 to 6'/min.). When the pen has returned to background, raise the pen, switch back to the original gamma range, lower the pen and continue logging the hole. A sample of a re-run is shown in figure 5c.

Section II (1000-C)

- 2-4.20 Upon reaching the top of the hole: 1. raise the pen, 2. set the brake, 3. switch the LOG SELECTOR to POWER OFF, 4. change the left pen to black (with the nib on the right) and remove the protective caps from both pens, 5. move the winch crank to the 1:1 ratio shaft, 6. switch the LOG SELECTOR to SP-R, 7. set the MV/DIV. and ohms/DIV. switch to 100, and the BIAS and DISPLACEMENT controls to zero. DO NOT shift the CHART DRIVE to OFF; the bi-directional chart drive will automatically follow the probe back downhole for the second run (SP and R).
- 2-4.21 Check the zero on both pens. The SP pen zero is on the left margin and the R pen zero is at the center of the chart. The scales and bias/displacement setting should be set on the trip downhole. The R pen will be off scale to the right until the water level is reached (when it comes back on scale). At this time you should start alternately lowering the OHMS/DIV. switch, to make the pen more active (larger fluctuations), and adjusting the DISPLACEMENT control to center the fluctuations on the right-hand half of the chart. The MV/DIV. switch and BIAS control are set in a similar manner; the POLARITY switch changes the polarity of the bias voltage to allow logging positive or negative SP. The SP log should cover the left-hand half of the chart and the R log the right-hand half.
- 2-4.22 You can now crank the probe back down the hole, setting the SP and R controls as you go. If the pens come into contact with each other, allow a few seconds for them to come back on their own, then if necessary, adjust the BIAS and/or the DISPLACEMENT controls. Turning the DISPLACEMENT control clockwise moves the R pen to the left; turning the BIAS control clockwise moves the SP pen to the right on "+" polarity and to the left on "-" polarity.
- 2-4.23 When you come out of the water the SP and R pens will rapidly go off scale. Raise the pens and turn the LOG SELECTOR to POWER OFF. The probe should be cranked out of the hole as fast as possible, to reduce time in the hole and the chance of getting stuck.
- 2-4.25 Retrieve the mud plug and wipe excess mud from it and the winch assembly. After storing the cable head and pulley assembly, tighten the cable slightly to prevent it from loosening on the drum and becoming tangled during shipping.
- 2-4.26 Make sure the LOG SELECTOR is in the POWER OFF position and the winch brake is on before storing or transporting the logger.

NOTE: All dimensions are in millimeters (inches)

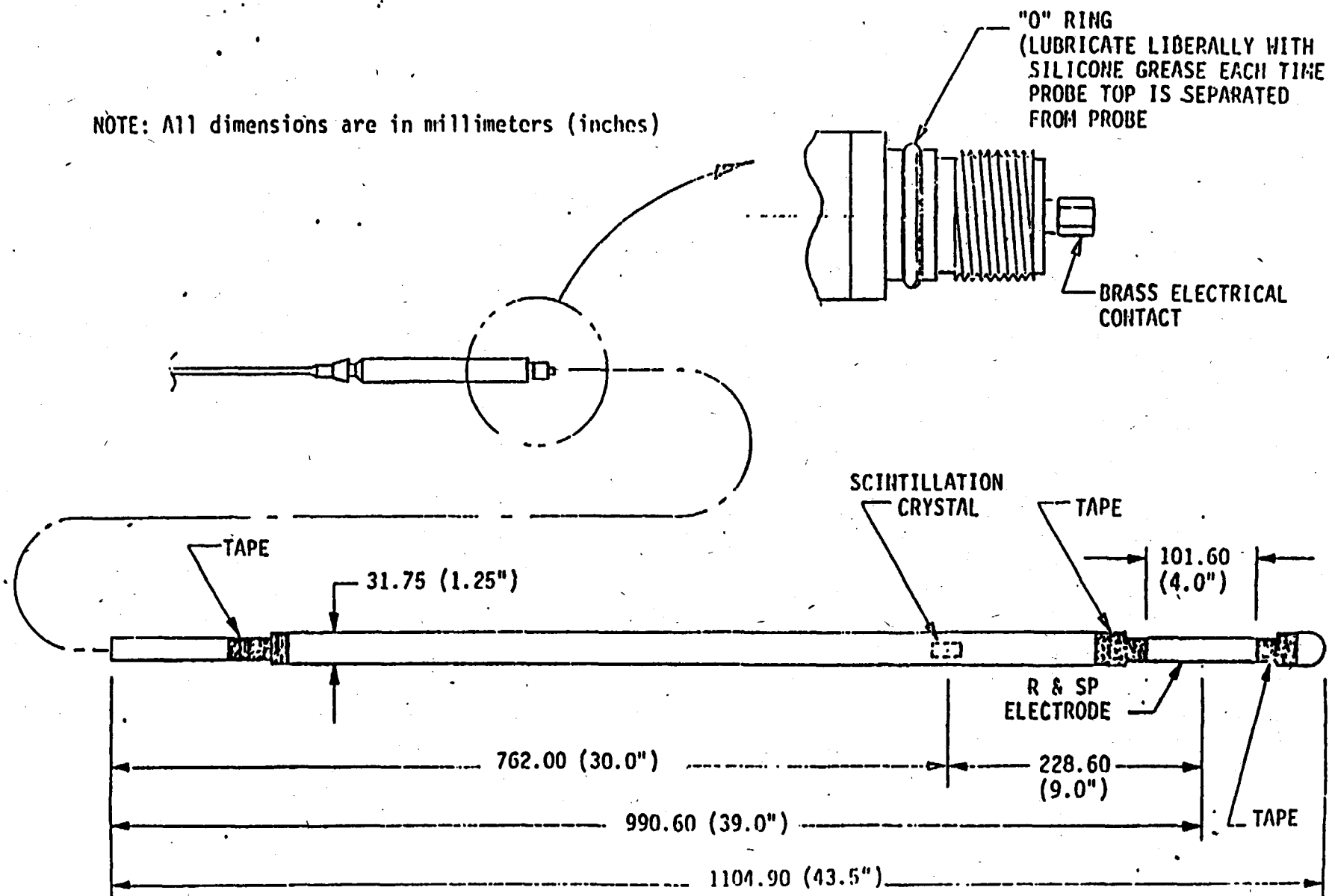
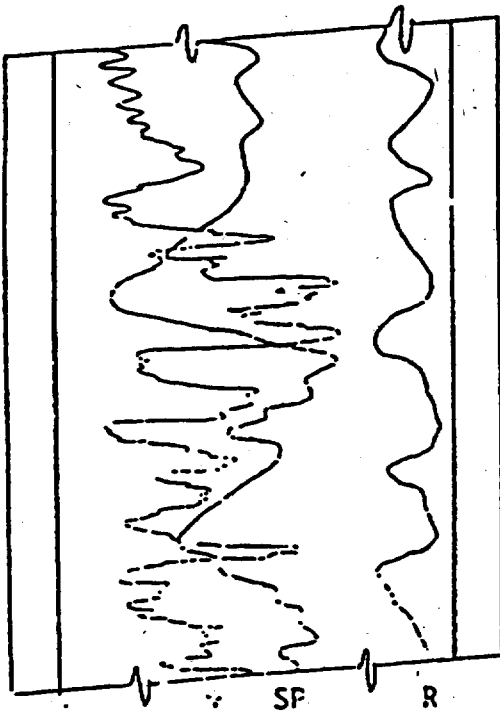
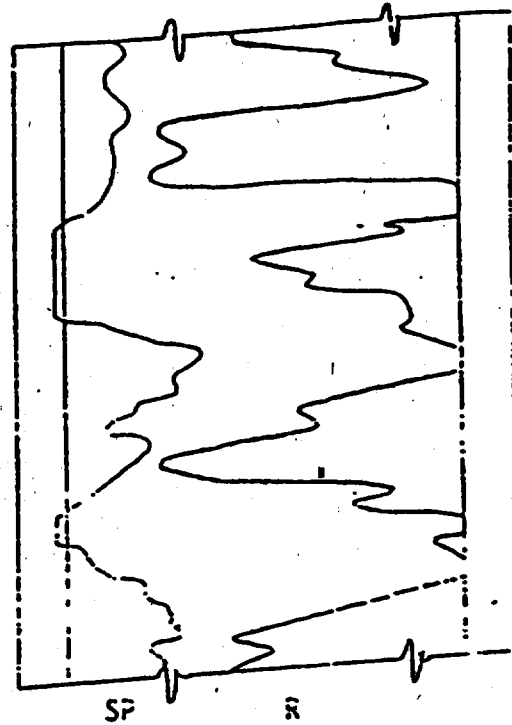


Fig. 4 PROBE DIMENSIONS

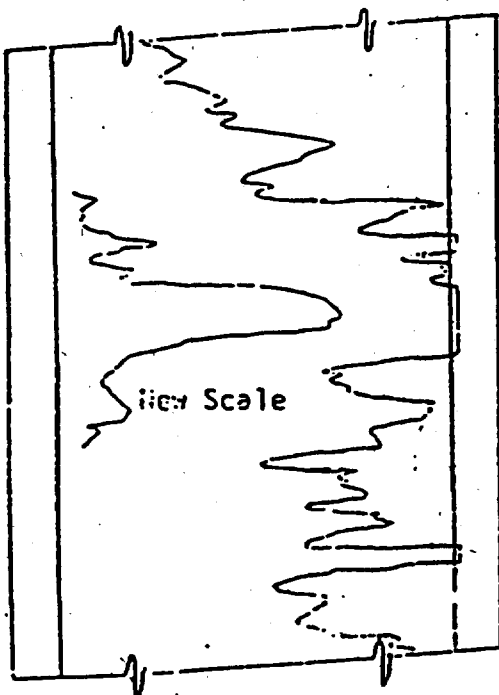
Section II (1000-C)



Example of a
good log
Fig. 5A



Incorrect Biasing
on the SP.
Range too high
on the R
Fig. 5 B



Re-Run
Fig. 5 C

Figure 5
Sample Logs

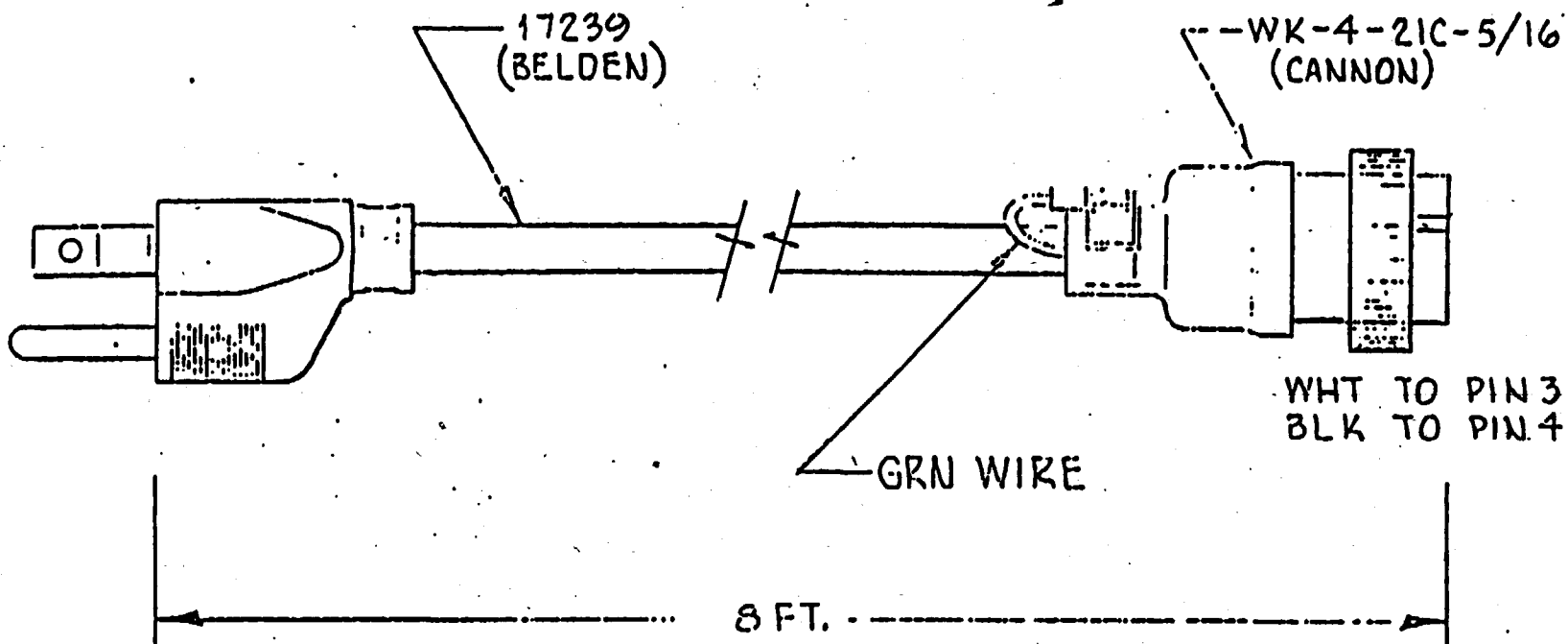
2-5.0 Operating Precautions

- 2-5.1 Keep the cable head threads and brass contact clean and the O-ring coated with silicone grease.
- 2-5.2 The probe top threads and area around the center connector must be kept free from dirt and grit.
- 2-5.3 If you become stuck in the hole DO NOT force the winch. If a moderate amount of pull from the winch will not free the probe a cable gripper and a jack or lever arm will be required.
- 2-5.4 When the CPS/DIV. switch is on 200 or above, the gamma DISPLACEMENT switch must be on 0 or the batteries will prematurely discharge.
- 2-5.5 DO NOT allow the probe to freewheel to the bottom of the hole, as damage may result to the photomultiplier tube and/or scintillation crystal.
- 2-5.6 In logging holes near the length of the cable, do not unspool cable completely. A minimum of 5 full wraps must be kept on the winch drum.
- 2-5.7 Keep the cable neat and evenly wound across the winch drum to prevent kinks and possible short-circuits.
- 2-5.8 Do not allow the cable to kink. This causes premature wear, and could cause a short between the center conductor and the steel armor.
- 2-5.9 Handle probe with care. Avoid rapid changes in temperature and sharp blows, especially in a lateral direction.
- 2-5.10 LOG SELECTOR must be in POWER OFF when changing or removing probes.

2-6.0 Battery Life & Re-charging

The life of a nickel cadmium battery is largely dependent on temperature; however, there are many other factors involved (discharge rate, type of charger, etc.). The cells used in the Model 1000-C nominally give 1000 complete charge-discharge cycles. The charging system used is of the constant current type, and can be left on for an indefinite period of time with no danger of overcharging. A sensor in the charger switches automatically for 120 or 240 volt a.c. operation. It is recommended that the unit be left on charge when it is not in use, and recharged at least every six months when in storage.

- 2-6.1 Connect the proper charging cable, for the voltage source available, to the BATTERY CHARGE connector. If the 12 vdc cable is used, the red lead connects to the positive (+) terminal of the source, and the black lead to the negative (-). If the a.c. cord is to be used, MAKE CERTAIN the CHARGING VOLTAGE SELECTOR SWITCH (located inside the chart access door) is in the PROPER POSITION. If used on 240 v. it will be necessary to cut the cord near the a.c. plug and install an appropriate connector. Refer to figure 6 for the proper connections. Connect the cable to the power source and observe the charge light (located inside the chart access door) come on to indicate proper charging. Should the charging indicator fail to light, check the connections to the power source, make sure the power source is active, and check the fuse (a.c. operation only) located inside the chart access door. NOTE: If the polarity of the 12 vdc cable is reversed, no damage will result, but the batteries will not charge.



Pin 4-----Black-----120 v. or 240 v. (Hot)
 Pin 3-----White-----120 v. or 240 v. (Return)
 Conn. Shell----Green-----Earth Ground

Pin 1----Black-----Common
 Pin 2----Red-----+12 to 14 v.d.c.

Fig. 6 Battery Charge Inputs

WATER FACTORS TO USE WITH
MOUNT SOPRIS MODEL S11, S11E, AND G-375 PROBES

<u>Hole Diameter</u>		<u>Correction Factor</u>
<u>in Inches</u>		
2.25		1.035
4.5		1.076
6.5		1.115
8.5		1.115

CASING FACTORS TO USE WITH
MOUNT SOPRIS MODEL S11, S11E, AND G-375 PROBES

<u>Casing Wall Thickness</u>		<u>Correction Factor</u>
<u>in Inches</u>		
1/16"		1.24
1/8"		1.33
3/16"		1.43
1/4"		1.62
3/8"		1.87

Gamma Time Constant (T.C.) Switch

This unit has been fitted with a Gamma time constant response switch. The switch is mounted on the dividing line between the Gamma and SP Range Switches. The switch has two positions, 2.4 and 0.3, corresponding to a full scale response time of 2.4 seconds and 0.3 seconds resulting from a full scale input step.

The switch should normally be set to the 2.3 position for gamma probes and to the faster 0.3 range for such measurements as temperature, caliper, flowmeter and fluid resistivity, etc. If more filtering is desired this last group of probes could be run with the 2.3 position. For the most accurate results the gamma calibrate potentiometer should be adjusted each time the T.C. switch position is changed. The effect of the T.C. switch positions can be seen by operating the 1000C in the Gamma position without a probe attached and switching between the 5K range and the Cal position for each T.C. position.

3 Theory of Operation

Figure 2 is a block diagram which shows the main elements of the HLP-2375 combination probe. Refer to the appropriate schematics located in Appendix 1 as necessary for the discussion of the operation checkout and trouble shooting of this probe.

As Figure 2 indicates, the active circuits of the HLP-2375 probe serve one function (i.e. a Gamma Ray Scintillometer).

The other functions of the probe, namely SP and resistance (R) are passive, consisting downhole only of the A electrode. Circuitry for these measurements is located in the HLM-2680 module which has been designed to complement the HLP-2375 probe.

3.1 Gamma Ray Scintillometer

The gamma ray scintillation circuit may be subdivided into two sections consisting of (1) the photomultiplier dynode power supply circuits and (2) the pulse detection shaping and line driving circuitry.

Photomultiplier Dynode Power Supply (See schematics for boards A2 and A3) - Unregulated power of at least 20 Vdc at the cablehead is required to operate the HLP-2375 probe. The photomultiplier dynode voltages are derived by stepping the basic 20 Vdc power supply up to a square wave of 120v (peak amplitude) through the multivibrator formed by Q1 and Q2 and transformer T1. The exact value of the AC voltage is deter-

the discriminator (adjustable through R6) the comparator will trigger causing transistor Q4 to saturate. The negative going pulse at the collector of Q4 triggers both one shots (U2). One of the one shots is used to drive the complementary emitter follower which drives a positive pulse of approximately 1 microsecond and 6.8 volts onto the cable. The other one shot is set at 5 microseconds and is used to electronically inhibit the first one shot from retriggering for that period of time. This introduces a known probe dead-time which is electronically compensated for in all Mt. Sopris ratemeters.

6 Service and Trouble Shooting

The HLP-2375 has one main active function and it should be a simple matter to isolate problems to either the natural gamma function, the uphole electronics, or cable. Before trouble shooting the probe, it is always wise to verify that the proper probe power (20 Vdc) is available between armor (common) and the center conductor (20 Vdc). The steps listed below outline a procedure for trouble shooting the HLP-2375 probe:

I. Preliminary Checks

- A. Check cable for shorts or leakage, and for opens (cable resistance).
- B. With probe connected and ratemeter module on, check between lines 1 and 2. There should be a DC voltage of 20 to 30 volts upon which some positive gamma pulses are riding.

1. If there is no DC voltage, then the problem is in the ratemeter.
 2. If the DC voltage is present, continue.
- C. Is the problem in the SP or resistance logs?

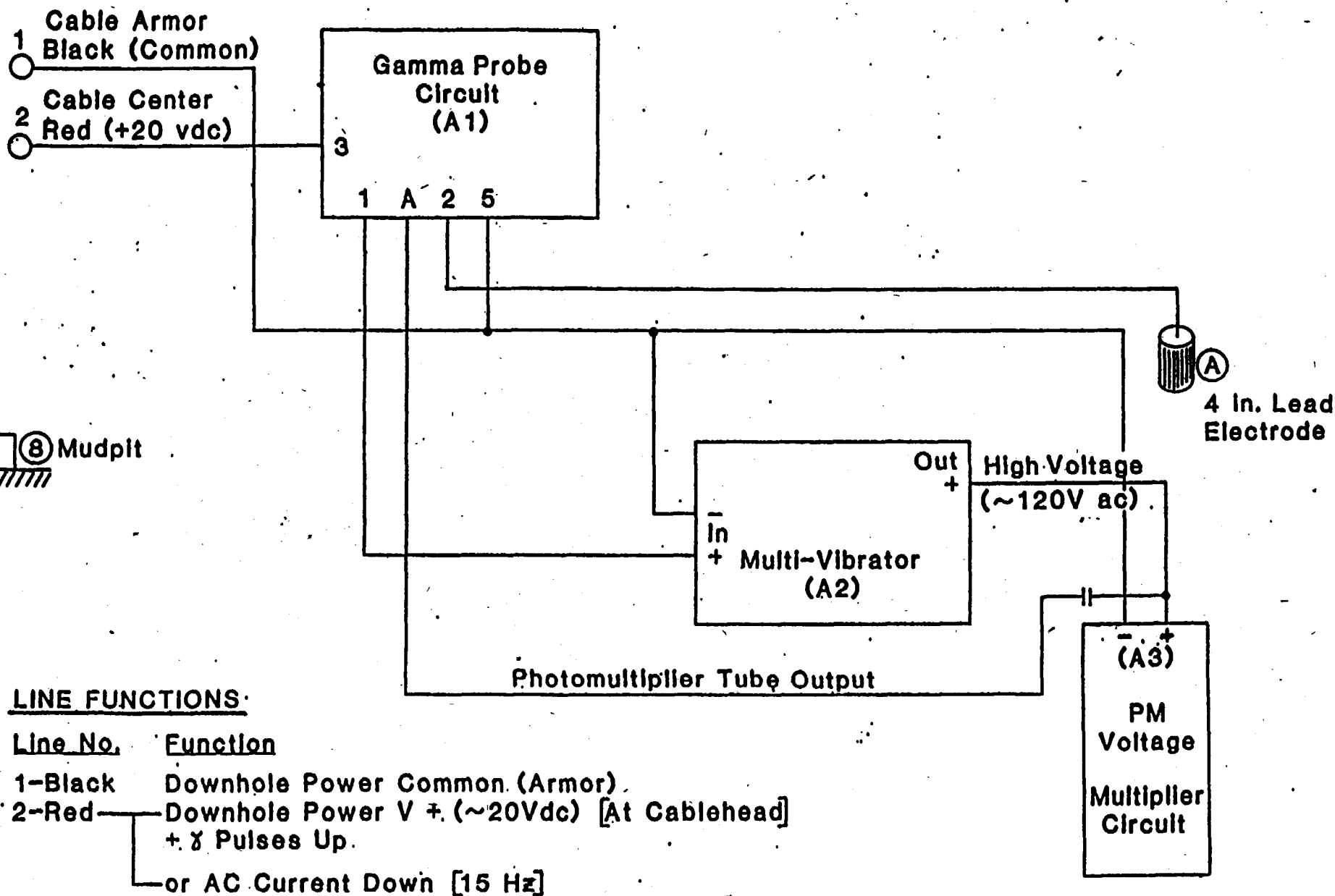
1. Yes - Check to see that when electrode A and the mud pit are connected through a power resistor, that a constant current sinewave is passing through the resistor.
 - a. If no current is passing in circuit, check for continuity between line 2 (center) and the 4 in. (A) lead electrode on the sonde. Also check for continuity between the mud pit electrode and pin K of the connector in the back of the module bin. If there is continuity but no current, the problem lies in the HLM-2680 module current generator.
 - b. If current signal appears to be correct but there is no resistance output, the problem still lies with the RESISTANCE circuit.
2. No - If there is a gamma problem and there is 20 to 30 Vdc between lines 1 and 2, then problem is downhole. Go to II.

(Note: Downhole power, lines 1 and 2, should be 30 VDC with probe disconnected and ratemeter on.)

II. Probe Checkout - Uphole OK

- A. Remove top sub on tool and carefully slide out the electronics section. After the electronics section is outside the housing, turn on the ratemeter.
 1. Verify that there is at least 20 Vdc between lines 1 and 2 (red and black wires) at the cablehead. If the voltage is less than this, there is either a bad downhole power supply (presumably, this possibility has been eliminated by now), or there's something loading the power supply.
 2. If the power supply is being loaded, check the gamma board.
- B. Gamma Board

1. If the problem lies with the gamma circuit and the H.V. is OK, remove all the IC's on the board. Check to see that the 17 Vdc supply is OK. If not, check for a bad regulator transistor Q7, or a diode D7. Also check Q5 and Q6 of the line driver for shorts or bad transistors.
 2. Check to see that there are random pulses at point A on the board. (It helps to have a small gamma source, but there should be enough background count to perform this check.)
 3. Follow the signal through Q1 and Q2, replacing these transistors if necessary.
 4. Replace each IC in turn. Note the 17 Vdc power and check to see that the pulses are properly amplified.
 5. Check for proper pulse widths on the one shots.
- C. Photomultiplier Power Supply Board
- Check the output of the high voltage board. Depending on the zener diodes selected, this should be a 500 Hz square wave with an amplitude of approximately 120v. This voltage should be rectified and multiplied at each successive dynode stage, with the lowest voltage being at dynode D10 and the highest (approximately 1400 Vdc) at the photocathode. If no output voltage is present across the secondary of T1, there is probably a faulty transistor Q1 or Q2 in the astable multivibrator circuit used to step up the basic 20 Vdc supply voltage.



MUDPIT CURRENT RETURN

FIGURE 2: FUNCTIONAL BLOCK DIAGRAM FOR THE HLP-2375 COMBINATION PROBE

Appendix A-A6

Calibration and Maintenance of the Flame-Ionization Detector (FID)

INSTRUMENT OPERATION PROCEDURE

**FOXBORO Model 128 Flame-Ionization Detector
For the Detection of Organic Vapors
Through Flame-Ionization**

January, 1991

SCOPE AND APPLICATION

The OVA 128 is a sensitive instrument designed to measure trace quantities of organic materials in the air. The instrument uses a hydrogen flame ionization detector with the sensitivity to measure in the parts per million (ppm) range (V/V) in the presence of atmospheric moisture, nitrogen oxides, carbon monoxide, and carbon dioxide. The instrument has a single linearly scaled readout from 0 to 10 ppm with a X1, X10, and X100 range switch.

The instrument measures organic vapor concentration by producing a response to an unknown sample, which can be related to a gas of known composition to which the instrument has previously been calibrated.

Reference:

Instruction Manual Model OVA 128, Century Organic Vapor Analyzer.

Apparatus:

OVA Model 128.

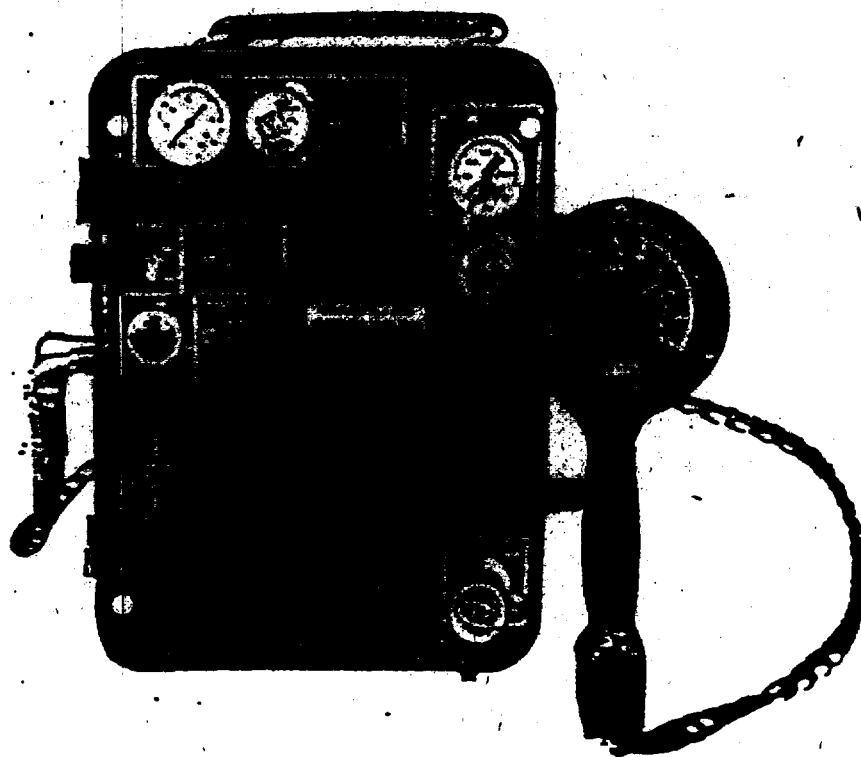
Battery Charger.

Side Pack Assembly and the Probe/Readout Assembly (See Figure 1).

Recorder.

Calibration Kit - Kit containing a gas cylinder of Zero air, a gas cylinder of 95 to 100 ppm Span Gas (Methane in air), and gas regulators.

Gas chromatographic columns.



ORGANIC VAPOR ANALYZER

FIGURE 1

1.0 CONTROLS AND INDICATORS

1.1 Side Pack Assembly

1.1.1 INSTR/BATT Test Switch - three position toggle switch controls all instrument electrical power except the pump and alarm power. It also permits the display of the battery charge condition on the readout meter.

1.1.2 PUMP (ON/OFF) Switch - toggle switch controls power to the internal pump and audio alarms.

1.1.3 IGNITER Switch - momentary push button switch connects the power to the igniter coil in the detector chamber and simultaneously disconnects power to pump.

1.1.4 CALIBRATE Switch (range selector) - selects the desired range: X1 (0 to 10 ppm); X10 (0 to 100 ppm); X100 (0 to 1000 ppm).

1.1.5 CALIBRATE ADJUST (zero) knob - potentiometer used to zero the instrument.

1.1.6 GAS SELECT KNOB (span control) - ten-turn dial readout potentiometer sets the gain of the instrument (commonly referred to as span control).

1.1.7 Recorder connector - five-pin connector used to connect the instrument to the recorder.

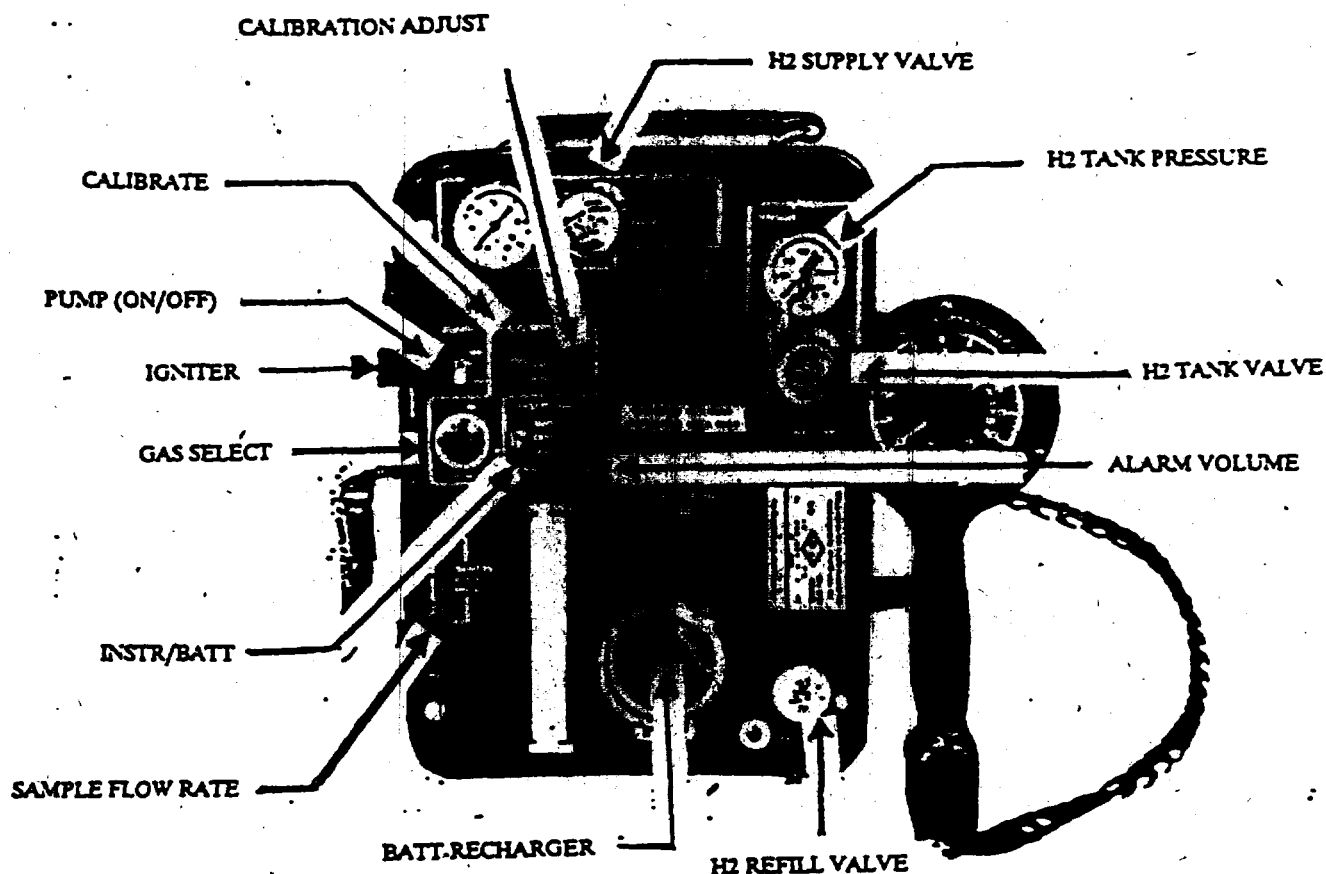
1.1.8 Charger connector - BNC connector used to connect the battery pack to the battery charger.

1.1.9 HYDROGEN TANK VALVE - valve used to supply or close off the fuel supply from the hydrogen tank.

1.1.10 HYDROGEN TANK PRESSURE indicator - high pressure gauge measures pressure in the hydrogen fuel tank which is an indication of fuel supply.

1.1.11 HYDROGEN SUPPLY VALVE - valve used to supply or close off hydrogen fuel to the detector chamber.

1.1.12 HYDROGEN SUPPLY PRESSURE indicator - low pressure gauge used to monitor hydrogen pressure at the capillary restrictor.



CONTROLS AND INDICATORS

FIGURE 2

1.1.14 REFILL CONNECTION - 1/4 in. AN fitting to connect the hydrogen refill hose to the instrument.

1.1.15 REFILL VALVE - valve to open one end of the instrument fuel tank for refilling with hydrogen.

1.1.16 **EARPHONE JACK** - used to connect the earphone.

1.1.17 **VOLUME knob** - potentiometer adjusts the volume of the internal speaker and earphone.

1.1.18 **Readout and sample connectors** - used to connect the sample hose and the cord from the probe/readout to the side pack.

2.0 STARTUP AND OPERATING PROCEDURES (Survey Mode)

2.1 Connect the Probe/Readout assembly to the sidepack assembly by attaching the sample line and electronic jack to the sidepack.

2.2 Select the desired sample probe and connect the probe handle. Before tightening the knurled nut, check that the probe accessory is firmly seated against the flat seals in the probe handle and in the tip of the telescoping probe.

2.3 Move the Instrument/Battery switch to the test position. The meter needle should move to a point beyond the white line, indicating that the battery has more than four hours of operating life before charging is necessary.

2.4 Move the Instrument/Battery switch to the "ON" position and allow a 5 minute warmup.

2.5 Turn the pump switch on.

2.6 Use the calibrate adjust knob to set the meter needle to the level desired for activating the audible alarm. If this alarm level is other than zero, the calibrate switch must be set to the appropriate range.

2.7 Turn the volume knob fully clockwise.

2.8 Using the alarm level adjust knob, turn the knob until the audible alarm is activated.

2.9 Move the calibrate switch to X1 and adjust the meter reading to zero using the calibrate adjust (zero) knob.

2.10 Open the hydrogen tank valve 1 or 2 turns and observe the reading on the hydrogen tank pressure indicator. (Approximately 150 psi of pressure is required for each hour of operation).

2.11 Open the hydrogen supply valve 1 or 2 turns and observe the reading on the hydrogen supply pressure indicator. The reading should be between 8 and 12 psi.

2.12 After approximately one minute, depress the igniter button until the hydrogen flame lights. The meter needle will travel upscale and begin to read "total organic vapors". **CAUTION:** Do not depress igniter for more than 6 seconds. If flame does not ignite, wait one minute and try again.

2.13 The instrument is ready for use.

2.14 NOTE: Use calibrate knob to "zero" out ambient background. For maximum sensitivity below 10 ppm, set calibrate switch to X1 and readjust the zero on the meter. To avoid false flame-out alarm indicator, set meter to 1 PPM with calibrate knob and make the differential readings from here. If the ambient background organic vapors are "zeroed out" using the calibrate adjust knob, the needle may move off-scale in the negative direction when the OVA is moved to a location with lower background.

3.0 SHUT DOWN

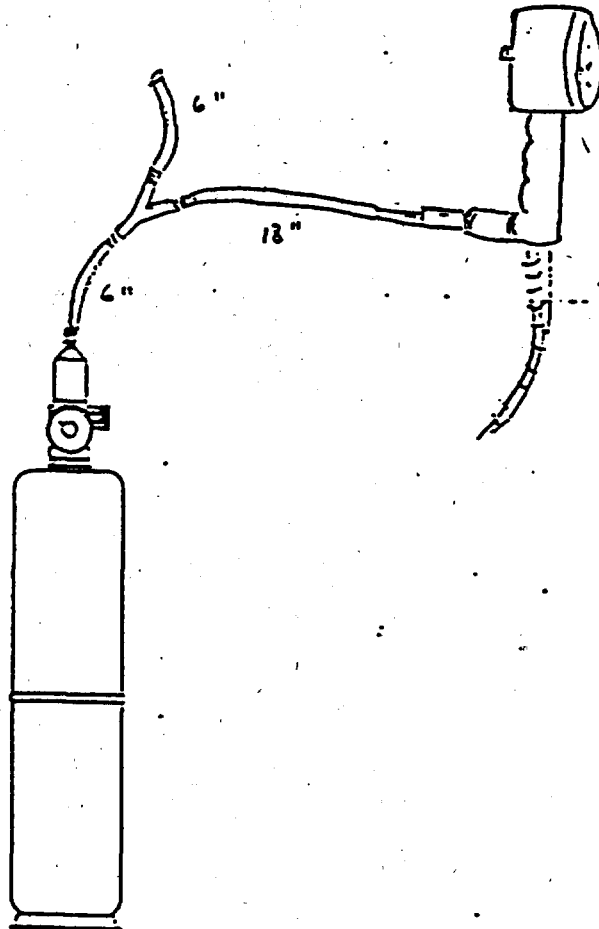
- 3.1 Close the hydrogen supply valve
- 3.2 Close the hydrogen tank valve
- 3.3 Move the instrument switch and pump switch to OFF.

4.0 CALIBRATION

Calibration by using a known methane concentration in air in the range of 95-100 ppm.

4.1 Attach one 6" piece of tubing to the regulator. The other 6" piece is the vent.

4.2 Attach the 18" branch length of tubing to the readout assembly. (See Figure 3).



CALIBRATION CYLINDER/PROBE ASSEMBLY

FIGURE 3

4.3 Place the instrument in normal operation and allow a minimum of 15 minutes for warm-up and stabilization.

4.4 Set the Gas Select control to 300. 30

4.5 Use the zero air cylinder and with the calibrate adjust zero knob adjust the meter reading to zero.

4.6 Place instrument in normal operation with calibrate switch set to X10 and gas select control set to 300.

4.7 Introduce a methane sample of a known concentration (between 90 and 100 ppm, not to exceed 100 ppm) and adjust trim pot R-32 so the meter reading corresponds to the known sample.

4.8 The instrument has been calibrated for methane with the panel mounted gain adjustment (gas select) set at reference 300.

4.9 Turn off hydrogen supply valve to put out the flame.

4.10 Leave calibrate switch on X10 position and use calibrate adjust (zero) knob to adjust meter reading to 4 ppm.

4.11 Place calibrate switch in X1 position and using trim pot R-31 adjust meter reading to 4 ppm.

4.12 Move calibrate switch to X10 position again. use calibrate adjust (zero) knob to adjust meter to a reading of 40 ppm.

4.13 Move calibrate switch to X100 position and use trim pot R-33 to adjust meter reading 40 ppm.

4.14 Move calibrate adjust (zero) knob to adjust meter reading to zero.

4.15 Unit is now balanced from range, calibrated to methane, and ready to use.

B

ANALYTICAL SERVICES STANDARD OPERATING PROCEDURES (SOPs)

- B1 Alkalinity**
- B2 Chloride and Sulfate**
- B3 Total Dissolved Solids**
- B4 Ammonia Nitrogen**
- B5 Nitrate & Nitrite Nitrogen**
- B6 Total Phenolics**
- B7 Grain Size Distribution**
- B8 Total Organic Carbon**
- B9 Hydraulic Conductivity**

B1

ALKALINITY

STANDARD
OPERATING
PROCEDURE

Subject or Title: Page 1 of 15

pH, Specific Conductance, and Alkalinity
(Automatic Titrator)

SOP No.: Revision No.: Effective Date:
LM-RMA-1071 1.0 August 8, 1990

Supersedes: Original

1. Scope and Application

1.1 Analytes

This method is to be used for the determination of pH, Specific Conductance, and Alkalinity. The different forms of alkalinity (total, bicarbonate, carbonate, and hydroxide) can also be calculated.

1.2 Applicable Matrices


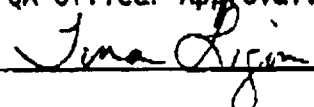
This method is applicable to drinking, surface and saline waters, domestic and industrial wastes.

1.3 Reporting Limits

The Enseco reporting limits are:
pH -- Detection limit not applicable
Specific conductance -- 1 umho/cm
Alkalinity -- 5 mg/L as Calcium Carbonate

1.4 Dynamic Range

This method is applicable to all ranges likely to be encountered. As a practical matter, samples with alkalinities greater than about 1200 mg/L as Calcium Carbonate require a smaller than normal volume or stronger titrant in order to keep the titrant volume to a reasonable amount.

Prepared by:	Date:
Lindsay Breyer/Roxanne Sullivan	5/24/88
Management Approval:	Date:
	8/6/90
QA Officer Approval:	Date:
	8/6/90

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1.5 Analysis Time

The approximate analytical time is five minutes per sample. Samples with high alkalinity or undissolved solids may require longer titration times.

2. Method Summary

2.1 Samples are analyzed on an automatic titrator.

2.2 The Specific Conductance is measured first by application of a voltage across the conductivity cell in series with a known reference resistance. The ratio of voltages across the cell and the known resistance is measured which allows the resistance of the cell to be calculated. This is then converted to the conductance of the cell. By calibrating against a solution of known conductance, the cell constant is determined and also the specific conductance of the sample in the cell. A thermocouple measures the temperature of the sample and electronically corrects the measurement to 25°C.

2.3 The pH is then determined electrometrically with a glass electrode in combination with a reference electrode. The special glass used in the electrode develops a voltage across it that depends on the pH of the solution being analyzed. The voltage is measured and converted to pH by calibration against buffers of known pH.

2.4 Alkalinity is determined by titrating the sample with a standardized acid to specified endpoints (pH 8.3 and 4.5). Alkalinity is calculated from the volume of acid required to reach the endpoints and is traditionally reported as Calcium Carbonate.

3. Comments

3.1 Interferences

- 3.1.1 Sodium can interfere with pH determinations at pH's higher than 10. This is controlled by using a "low sodium error" electrode.
- 3.1.2 Oil and particulate matter in the samples may coat the pH electrode and conductivity cell causing erroneous readings. This will also affect the alkalinity results.

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- 3.1.3 Temperature variations will affect both pH and conductance measurements. The conductance measurements are corrected for temperature automatically. The effect on pH is controlled by performing the measurements at the same (i.e. room) temperature. All samples must be within 20°C.
- 3.1.4 Salts of weak organic and inorganic acids will contribute to alkalinity. If the alkalinity is intended to be a measure of carbonate and bicarbonate only, the presence of these substances will cause high results.
- 3.1.5 The alkalinity is titrated to pH 4.5. This endpoint may not be appropriate for all samples.
- 3.1.6 Specific conductance is extremely sensitive to contamination. The titration head, electrodes, and all glassware must be thoroughly rinsed with deionized water to remove all traces of salts.
- 3.1.7 Samples not in equilibrium with the atmosphere may exhibit changes in pH and in the distribution of the various forms of alkalinity when exposed to the atmosphere. The sample containers should be filled completely and kept closed until just prior to the analysis. The analysis should be performed as soon as possible.
- 3.1.8 The pH electrode may exhibit slow or noisy response with high purity waters due to the lack of ionic strength.

4. Safety Issues

- 4.1 All employees are expected to be familiar with and follow the procedures outlined in the Enseco-RMAL safety plan. Lab coats and safety glasses are required in all laboratory areas at all times. If you have any questions or safety concerns, see your supervisor or safety officer.
- 4.2 Normal laboratory safety precautions should be followed. There are no special concerns with this method.

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5. Sample Collection, Preservation, and Holding Times

- 5.1 Samples are to be collected in plastic bottles and refrigerated to 4°C.
- 5.2 Samples for alkalinity must not be filtered, diluted, concentrated, or altered in any way.
- 5.3 If the conductance measurement cannot be performed within 24 hours of collection, the sample should be filtered.
- 5.4 The holding time for these parameters will be considered to be 24 hours since all are analyzed at once. The official holding times are:

pH -- 24 hours
Conductance -- 28 days
Alkalinity -- 14 days

6. Apparatus

- 6.1 Radiometer Automatic Titration System, consisting of autosampler, burette, pH meter and electrodes, conductance meter and cell, temperature probe, stirrer, printer, etc.
- 6.2 Plastic beakers to fit autosampler.
These must be thoroughly rinsed to remove all traces of salt.
- 6.3 Oxford pipette calibrated to 10.0 mL, and disposable tips.
- 6.4 Miscellaneous laboratory apparatus and glassware.

7. Reagents and Standards

- 7.1 pH Buffers, 4.0, 7.0, and 10.0
Obtained commercially
- 7.2 0.01 M Potassium Chloride (Conductance Standard)

Keep a beaker full of KCl in a 105°C oven for routine use. Dissolve 0.7456 g in deionized water and dilute to 1 liter. This solution has a conductance of 1417 $\mu\text{mho/cm}$ at 25°C. Prepare monthly.

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August 8, 1990**7.3 0.0500 N Sodium Carbonate**

Dry anhydrous sodium carbonate at 250°C for four hours and cool in a desiccator. Dissolve 2.6497 g in deionized water and dilute to 1 liter.

7.4 0.1 N Sulfuric Acid (Strong Alkalinity Titrant)

Very carefully add 3.0 mL concentrated sulfuric acid to approximately 500 mL deionized water and dilute to 1 liter. This solution must be standardized using the procedure given in section 8. Record standardization data and obtain a verification number from the Standards Prep Department.

7.5 0.02 N Sulfuric Acid (Alkalinity Titrant)

Very carefully add 3.0 mL concentrated sulfuric acid to 5 liters of deionized water. Standardize using the procedure given in section 8. Record standardization data and obtain a verification number from the Standards Prep Department.

7.6 Saturated NaCl

Dissolve 35.7 g of NaCl in 100 mL of cold DI.

8. Procedure**8.1 Preparation**

8.1.1 Thoroughly homogenize samples before taking an aliquot for analysis. Allow aliquots to equilibrate to room temperature before analyzing.

8.2 Instrument Set-up and Calibration**8.1.2 Standardization of Alkalinity Titrant**

Accurately measure 0.05 N Sodium Carbonate into a 250 mL beaker. Use 40 mL if 0.1 N Sulfuric Acid is being standardized or 15 mL for 0.02 N Sulfuric Acid. Add deionized water to bring the total volume to 100 mL.

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Titrate with the appropriate strength acid to a pH of about 5. Lift electrode and rinse into beaker. Cover with a watch glass and boil solution gently for 3 - 5 minutes. Cool to room temperature, rinse watch glass into beaker, and continue the titration to the pH inflection point. Calculate the acid normality using the formula in section 10.

Perform all titrations for standardization in duplicate.

8.2.2 Calibration for pH

Be sure that the reference electrode has been filled with saturated potassium chloride and that all electrodes and the titration head are clean and dry (only use Kim wipes to dry the electrodes).

Pour approximately 20 mL pH 7 buffer into a beaker and lower the electrodes into it. Place the titrator in pH mode and press the CAL button when the reading has stabilized. Step through the calibration routine by pressing the CAL button. When Buffer 2 is displayed, rinse the electrodes and immerse in pH 4 buffer. Allow the reading to stabilize and continue with the calibration routine. At each step, make sure that the displayed parameter is correct (e.g. buffer value, temperature, etc.). If you need to change an entry, press the SHIFT button and enter the correct value. When Buffer 3 is displayed, press the pH button to exit the calibration routine (this part of the calibration routine is for a different application not used here). Rinse the electrodes.

8.2.3 Calibration for Conductance

Be sure that the electrodes and titration head are clean and dry (only use Kim wipes to dry the electrodes).

Pipette 20 mL Conductance Standard (0.01 M potassium chloride) into a clean beaker and immerse the electrodes. Verify that the reference temperature is set to 25°C and

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the temperature compensation is 2% per degree C. Then adjust the cell constant to give a reading of 1.42 mS/cm. Rinse the electrodes when done. Since the accuracy of this measurement is very dependent upon electrode position it might be advised to calibrate on the first two cups of an autotitrator run. The position of the arms and electrodes are consistent during a run. Calibration should be done daily.

8.3 Analysis

- 8.3.1 Obtain samples from sample receiving. Check each sample for special instructions. Make copies to put into your data package. Fill out the benchsheet.
- 8.3.2 Turn on the power to the burette and autosampler. Be sure the titrant reservoir is full. The 0.02 N Sulfuric Acid should be used. If new titrant has been prepared, rinse out the reservoir with it, refill, and flush out the burette and titrant delivery line. Be sure that there are no bubbles in the line.
- 8.3.3 Set the autosampler to 2 rinses and a delay of 0.4 minutes. Fill two beakers with deionized water (20 - 25 mL) and place them in positions 19 and 20. These are the rinse beakers.
- 8.3.4 Set the titrator to EP mode and be sure that Method 1 is displayed.
- 8.3.5 Pipette 20 mL of each sample into a clean beaker and place on the turntable. A 10 mL Oxford pipette may be used to dispense two 10 mL aliquots of sample, if it has been calibrated previously to 10 mL. Record the sample ID and position on the titrator. Allow sample aliquots to equilibrate to room temperature.
- 8.3.6 Press START NEW CYCLE on the autosampler and the analysis will commence. Results will be automatically printed out and the sampler will stop when all samples have been titrated. At this point, additional samples can be placed on the titrator and analyzed starting at step 8.3.4. Be sure to keep the titrant reservoir filled and replace the water in the rinse beakers periodically.

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8.3.7 The titrator has been programmed to deliver a maximum volume of 25 mL titrant. Samples requiring more than this should be reanalyzed using a smaller aliquot or strong titrant (0.1 N Sulfuric Acid). See your supervisor for the corrections to be made in the program for titrator.

8.3.8 Samples with a conductance greater than 50,000 umho/cm need to be run on the high curve. See Appendix 1.0.

8.4 Conclusion

8.4.1 Clean up the work area.

8.4.2 Return samples to Sample Receiving.

8.4.3 Calculate the results according to the calculation section below.

8.4.4 Complete the data package making sure it is filled out properly and all the results are highlighted. Be sure to include the tape and copies of the special instructions. Submit the data package to your supervisor for approval.

9. QA/QC Requirements

9.1 QC Samples

9.1.1 Deionized water and tap water are to be analyzed at least once daily. The conductance of these is to be recorded in the notebook provided for that purpose.

9.1.2 Check a 10.0 pH standard with each new calibration.

9.1.3 One blank and DCS samples must be analyzed in every batch of 20 or less samples. The blank is deionized water.

9.1.4 A blank and standard check are to be performed after every 10 or less readings and at the end of the run. Use the DCS for standard checks. The blank is deionized water.

9.1.5 Duplicates may be required as project specific QC.

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- 9.1.6 Spikes for total alkalinity may be required for project specific QC. Spikes are made by adding 1.0 mL 0.05 N Sodium Carbonate to 20 mL sample. The spiking concentration is 2500 x Normality of Sodium Carbonate.

It is not possible to spike pH, conductance, or the individual forms of alkalinity.

9.2 Acceptance Criteria

- 9.2.1 Deionized water should have a conductance of 2 umho/cm or less. The alkalinity should be less than 5 mg/L. The pH is usually 5 - 6.
- 9.2.2 There are no acceptance criteria for tap water. Unusual readings should be reported to your supervisor.
- 9.2.3 pH 10.0 standard should read within $\pm 2\%$ of 10.0.
- 9.2.4 Control limits for accuracy and precision (RPD) on DCS samples are:

Parameter	Accuracy	Precision
pH	98 - 102%	5%
Conductance	95 - 105%	5%
Alkalinity	90 - 110%	10%

- 9.2.5 Standard check should be within 10% of the expected value.
- 9.2.6 Blanks should be less than the 2x reporting limits for conductance and alkalinity pH blanks should be around 5 to 6.
- 9.2.7 There are no acceptance criteria for project specific QC.

9.3 Corrective Action Required

- 9.3.1 Follow the corrective actions outlined in the current Enseco QAPP manual.

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- 9.3.2 If the deionized water has a conductance more than 2 $\mu\text{mho}/\text{cm}$, repeat the reading. If it is still high, the deionized water system may need maintenance. Notify your supervisor.
- 9.3.3 If the pH 10.0 standards is not within limits, recalibrate and check the standard again. If it is still out of limits, see your supervisor.
- 9.3.4 If the control limits for the DCS samples are exceeded, the problem must be corrected and the samples reanalyzed. Check the calibration procedures and thoroughly clean the electrodes, titration head, and beakers. The most common problem is contamination, which causes high conductance readings.

10. Calculations

10.1 Standardization of Alkalinity Titrant

$$\text{NACID} = \text{NBASE} \times \text{VBASE} / \text{VACID}$$

NACID = Normality of titrant

NBASE = Normality of Sodium Carbonate

VBASE = Volume of Sodium Carbonate titrated, mL

VACID = Volume of titrant needed for titration, mL

10.2 pH values are recorded directly from the titrator printout.

10.3 Conductance values are taken as-is from the print out. If they are printed out in mS/cm , they must be multiplied by 1000. The units uS/cm are equivalent to $\mu\text{mho}/\text{cm}$.

10.4 Three values for alkalinity will be printed if the pH is greater than 8.3. Record the values at pH 8.3 and 4.5 on the bench sheet. The third value is the difference and is ignored. Note that some results will be printed in scientific notation; be careful to check this when recording results.

If the pH is less than 8.3, only one value will be printed (at pH 4.5). Record this value on the bench sheet. P alkalinity is ND on these samples.

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Calculate the individual forms of alkalinity:

 $T = \text{Total Alkalinity} = \text{Alkalinity at pH 4.5}$
 $P = \text{P Alkalinity} = \text{Alkalinity at pH 8.3}$

	Hydroxide Alkalinity	Carbonate Alkalinity	Bicarbonate Alkalinity
$P = \text{ND}$	ND	ND	T
$P < T/2$	ND	$2 \times P$	$T - 2 \times P$
$P = T/2$	ND	T	ND
$P > T/2$	$2 \times P - T$	$2(T - P)$	ND
$P = T$	T	ND	ND

Record the values for Total, Bicarbonate, Carbonate, and Hydroxide Alkalinity.

11. Reporting Requirements

11.1 Units

- 11.1.1 pH has units of "units".
- 11.1.2 Conductance is reported as $\mu\text{mho/cm}$ at 25°C .
- 11.1.3 All forms of alkalinity are reported in mg/L as Calcium Carbonate.

11.2 Limits

- 11.2.1 All values of pH are reported as measured.
- 11.2.2 Conductance results less than $1 \mu\text{mho/cm}$ are reported as ND.
- 11.2.3 Alkalinity results less than 5 mg/L are reported as ND.
- 11.2.4 All reporting limits must be multiplied by any prep or analytical dilution.

11.3 Significant Figures

- 11.3.1 pH is reported to one decimal place.

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11.3.2 Significant figures:

<u>Result</u>	<u>Significant Figure</u>
<10	2
10	3
>10	3

11.4 LDMS data entry

11.4.1 The usual standards for data entry apply.

11.5 Anomalies

Document any non-standard procedures or anomalies.

Some common anomalies are:

1. Missed holding time.
2. Wrong bottle type used.

12. Review Requirements

12.1 All data packages must be complete. They must be properly signed and dated. See attached example.

12.2 Confirm that special instructions were appropriately observed.

12.3 Check the calculations. Make sure all dilution factors were properly applied.

12.4 Check the reporting limits. Remember that the reporting limits must be multiplied by any dilution factor.

12.5 Make sure all anomalies are documented.

12.6 TDS/COND ratio should fall between 0.55 and 0.81.

**pH, Specific Conductance, and Alkalinity
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August 8, 1990**13. References****13.1 Source Method**

- 13.1.1 pH is EPA Method 150.1.
- 13.1.2 Conductance is EPA Method 120.1.
- 13.1.3 Alkalinity is EPA Method 310.1.

13.2 Related Documents

- 13.2.1 Additional information was obtained from Standard Method #403 for the alkalinity procedure.
- 13.2.2 Enseco QAPP.
- 13.2.3 M-EQA-0002 Internal QC Checks--Laboratory Performance QC.
- 13.2.4 LM-RMA-1003 Alkalinity (Manual Titration)
- 13.2.5 LM-RMA-1047 pH (soils and wastes)

13.3 Deviations from Source Methods and Rationale

- 13.3.1 Successive aliquots of sample are not analyzed for pH as in the reference methods.
- 13.3.2 Temperature is not reported with the pH measurement. All measurements are made at room temperature (approximately 22°C).
- 13.3.3 Alkalinity titrations are not performed at 25°C as specified in the reference method.
- 13.3.4 Method 310.1 contains no provisions for determining the different forms of alkalinity. This part of the method was obtained from Standards Methods for the Examination of Water and Wastewater, 14th Edition.

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13.3.5 A fixed endpoint of 4.5 is used for all alkalinity titrations.

13.4 Updates to SOP (Original to Version 1.0)

1. Format updated.
 2. Significant figures changed.
 3. Appendix 1.0 added for high conductance curve.
 4. pH 10.0 standard will be checked with each calibration curve.
-

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Appendix 1.0 High Conductivity Curve

1. Calibrate on saturated NaCl. The cell constant should be adjusted so the conductivity of the NaCl reads 251,000 umho/cm.
2. There is no QC.
3. Run your samples. Run DI rinses between each sample and have a double rinse cycle.

ANALYST RS
DATE/LOT 06 AUG 90 R
SPIKE

PH. 9.0 / _____
COND. 1130 / _____
ALK. 300 / _____

TITRANT: 0.025 N

** TOTAL PAGE.021 **

B2

CHLORIDE AND SULFATE

Subject or Title:

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Anion Analysis by Ion Chromatography

SOP No.:
LM-RMA-1025Revision No.:
3.0Effective Date:
1/25/93Supersedes:
2.0 (12/17/90)

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1. Scope and Application

1.1 Analytes

This method is applicable to the determination of the following inorganic anions:

Fluoride	Nitrate (as N)
Chloride	
Bromide	Sulfate

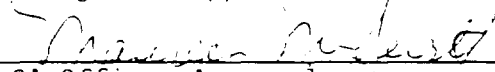

Prepared by:
Eileen BurkeDate:
January 25, 1993

Management Approval:

Date:

QA Officer Approval:

Date:


2/1/93
3/1/93

Anion Analysis by Ion Chromatography

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1.2 Applicable Matrices

1.2.1 This method is applicable to drinking and surface waters as well as mixed domestic and industrial wastewaters.

1.2.2 The method may be applied to nonaqueous matrices by analyzing a deionized water leachate of the sample (SOP LM-RMA-1074 D.I. Leach).

1.3 Reporting Limits

Enseco's standard reporting limits are:

<u>Analyte</u>	<u>Water (mg/L)</u>
Fluoride	0.1
Chloride	3
Bromide	0.2
Nitrate (as N)	0.1
Sulfate	5

Lower reporting limits may be available under special conditions. Consult the laboratory for details.

Nonaqueous reporting limits, when determined using DI leach SOP LM-RMA-1074, will be a factor of 5 higher than the above aqueous limits.

1.4 Dynamic Range

The dynamic range of the method is anion specific as indicated in the following table and can be extended by diluting the sample:

<u>Analyte</u>	<u>Range (mg/L)</u>	<u>Approximate Retention Times</u>
Fluoride	0.1 - 50	1.0
Chloride	3 - 300	1.6
Bromide	0.2 - 50	2.8
Nitrate	0.1 - 50	3.2
Sulfate	5 - 500	6.5

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1.5 Analysis Time

The approximate analysis time for water samples is 10 minutes, not including data reduction. Nonaqueous samples require a DI leach, whose approximate preparation time is 2 hours.

- 1.6 This method is restricted to use by or under the supervision of analysts experienced in the use of ion chromatography and in the interpretation of the resulting ion chromatogram. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 9.1

2. Method Summary

A small volume of sample is introduced into the Dionex ion chromatograph. The anions of interest are separated and measured using a system comprised of a guard column, separator column, suppressor column, and conductivity detector. Results are processed using Dionex software.

3. Comments

3.1 Interferences

3.1.1 Interferences can be caused by substances with retention times that are similar to and overlap those of the anion of interest. Large amounts of an anion can interfere with the peak resolution of an adjacent anion. Sample dilution and/or spiking can be used to solve most interference problems.

3.1.2 Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baseline in ion chromatograms.

3.1.3 Samples that contain particles larger than 0.45 microns and reagent solutions that contain particles larger than 0.20 microns require filtration to prevent damage to instrument columns and flow systems.

3.2 Helpful Hints

3.2.1 The water dip or negative peak that elutes near, and can interfere with, the fluoride peak can be eliminated by the

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addition of the equivalent of 1 mL of concentrated eluent (7.3 100X) to 100 mL of each standard and sample.

- 3.2.2 Large concentrations of any one ion (such as chloride in brines) may affect the retention time of the other ions and mask peaks.
- 3.2.3 Molybdate has the same retention time as sulfate; nitrite elutes very close to chloride.
- 3.2.4 This method is not recommended for leachate samples when acetic acid is used for pH adjustment. The retention times of the anions differ when large amounts of acetate are present.
- 3.2.5 This method is not recommended for fluoride. Any anion that is not retained by the column will elute in the area of fluoride and interfere.

4. Safety Issues

- 4.1 All employees are expected to be familiar with and follow the procedures outlined in the Enseco-RMAL safety plan. Lab coats and safety glasses are required in all laboratory areas at all times. If you have any question or safety concerns, see your safety officer.
- 4.2 Normal accepted laboratory safety practices should be followed during reagent preparation and instrument operation.

5. Sample Collection, Preservation, and Holding Times

- 5.1 Samples should be collected in scrupulously clean glass or polyethylene bottles.
- 5.2 Samples are to be preserved and analyzed in accordance with the following:

Analyte	Preservation	Holding Time
Fluoride	40C	28 Days
Chloride	40C	28 Days
Bromide	40C	28 Days
Nitrate (as N)	40C	48 Hrs
Sulfate	40C	28 Days

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6. Apparatus

- 6.1 Balance - Analytical, capable of accurately weighing to 0.0001 g.
- 6.2 Ion chromatograph - Analytical system complete with ion chromatograph and all required accessories.
 - 6.2.1 Anion guard column: AG4A, 4X50mm, Dionex P/N 37042.
 - 6.2.2 Anion separator column: AS4A, 4X250MM, Dionex P/N 37041.
 - 6.2.3 Anion micromembrane suppressor (AMMS): Dionex P/N 38019.
 - 6.2.4 Detector - Conductivity cell: approximately 6 uL volume.
- 6.3 Sample bottles: Glass or polyethylene of sufficient volume to allow replicate analyses of anions of interest.
- 6.4 Miscellaneous laboratory glassware and pipettes.

7. Reagents and Standards

- 7.1 Reagent water: Distilled or deionized water, free of the anions of interest and containing particles no larger than 0.20 microns.
- 7.2 Eluent solution: Sodium bicarbonate (CAS RN 144-55-8) 0.0018M, sodium carbonate (CAS RN 497-19-8) 0.0017M. Dissolve 0.286 g sodium bicarbonate (NaHCO_3) and 0.382 g of sodium carbonate (Na_2CO_3) in reagent water and dilute to 2 liters.
- 7.3 Regeneration solution (AMMS): Sulfuric acid (CAS RN 7664-93-9) 0.025N. Dilute 2.8 mL 50% sulfuric acid (H_2SO_4) to 2 liters with reagent water.
- 7.4 Stock standards: May be purchased as certified solutions or prepared from ACS reagent grade materials (dried at 105°C for 30 min) as listed below; may be prepared individually or as one stock standard.
 - 7.4.1 Fluoride (F) 1000 mg/L: Dissolve 2.2100 g sodium fluoride (NaF CAS RN 7681-49-4) or 3.0580 g potassium fluoride in reagent water and dilute to 1 L in a volumetric flask.

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- 7.4.2 Chloride (Cl) 1000 mg/L: Dissolve 1.6485 g sodium chloride (NaCl CAS RN 7647-14-5) in reagent water and dilute to 1 L in a volumetric flask.
- 7.4.3 Bromide (Br) 1000 mg/L: Dissolve 1.2878 g sodium bromide (NaBr CAS RN 7647-15-6) or 1.4893 g potassium bromide (KBr) in reagent water and dilute to 1 L in a volumetric flask.
- 7.4.4 Nitrate (NO₃-N) 1000 mg/L: Dissolve 6.0681 g sodium nitrate (NaNO₃ CAS RN 7631-99-4) or 7.2182 g potassium nitrate in (KNO₃) reagent water and dilute to 1 L in a volumetric flask.
- 7.4.5 Sulfate (SO₄) 1000 mg/L: Dissolve 1.8141 g potassium sulfate (K₂SO₄ CAS RN 7778-80-5) in reagent water and dilute to 1 L in a volumetric flask.

These stock standards are stable for at least one month when stored at 40°C. Dilute working standards should be prepared weekly.

- 7.5 Secondary standards: Make up individual secondary standards for each anion in 100 mL volumetric flasks as follows:

Analyte	Concentration (mg/L)	Stock Std. (mLs)	Final Volume (mLs)
Fluoride	100	10	100
Bromide	100	10	100
Nitrate	100	10	100
Chloride	100	10	100
Sulfate	100	10	100

- 7.6 Stock DCS solutions, 1000 ppm.

Stock DCS solutions may be purchased as certified solutions or prepared from ACS reagent grade materials (dried at 105° for 30 minutes) obtained from standards prep as listed below. The stocks may be prepared individually or as one stock DCS. 1000 mg/L DCS stock solutions should be made from a different source than the calibration stocks. If the stock standards are purchased, the concentration of the analytes purchased will be 10 times the final concentration of the working DCS standards. A 10X dilution will be required for purchased stock standards.

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7.7 Working DCS solution

Dilute down the stock DCS solutions (1000 ppm) into one 100 mL volumetric as follows:

<u>Analyte</u>	<u>Final Concentration (mg/L)</u>	<u>Stock Stds. (mLs)</u>
Fluoride	20	2
Chloride	200	20
Bromide	20	2
Nitrate	20	2
Sulfate	200	20

7.8 CCV Solution

Prepare a CCV solution at the following concentrations from the stock std. solutions in a 100 mL volumetric.

<u>Analyte</u>	<u>Concentration (mg/L)</u>	<u>Stock Std. (mLs)</u>
Fluoride	10	1
Chloride	100	10
Bromide	10	1
Nitrate	10	1
Sulfate	100	10

7.9 All preparations of calibration standards and reagents shall be documented in the IC Standard/Reagent Logbook. The information required shall include: reagent used, brand, lot number, expiration date, amount used, final volume, final concentration, analyst preparing, date, and expiration date of standard prepared.

7.10 Each stock standard should be verified before use and given a unique verification number. Working dilutions shall also be documented in the IC Standard/Reagent Logbook. The information required shall include: verification number of stock(s), expiration date of stock(s), amount used, final volume, final concentration, analyst name, and date made.

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8. Procedure

8.1 Preparation

- 8.1.1 Obtain samples from sample receiving. Check each sample for special instructions making copies to put along with the data package. Fill out your benchsheet.
- 8.1.2 Unless particulate matter is present, water samples are analyzed directly without advanced preparation. Samples containing particulate matter must be filtered through a 0.45 μ m filter prior to analysis.
- 8.1.3 Soil samples are prepared by leaching one part sample with five parts deionized water.
See SOP: LM-RMA-1074 D.I. Leach.

8.2 Instrument Set-up and Calibration

- 8.2.1 Establish the following ion chromatographic operating parameters:

Columns - AS4A Separator
AG4A Guard
Anion Micromembrane Suppressor

- 8.2.2 Prepare a series of standards at the following concentrations. Each standard should be made up in 100 mL volumetric with deionized water. This is the normal operating curve for RMA. Other curves can be made to meet client request.

Std. 1 is a eluent blank

<u>Std. 2</u>	<u>Concentration</u> (mg/L)	<u>Standard</u> <u>to be diluted</u> (ppm)	<u>Stock</u> (mLs)	<u>Final</u> <u>Volume</u> (mLs)
Fluoride	0.1	100(Secondary)	0.1	100
Chloride	1	1000(Stock)	0.1	100
Bromide	0.1	100(Secondary)	0.1	100
Nitrate	0.1	100(Secondary)	0.1	100
Sulfate	1	1000(Stock)	0.1	100

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<u>Std. 3</u>	<u>Concentration</u> (mg/L)	<u>Standard</u> <u>to be diluted</u> (ppm)	<u>Stock</u> (mLs)	<u>Final</u> <u>Volume</u> (mLs)
Fluoride	1	1000	0.1	100
Chloride	10	1000	1.0	100
Bromide	1	1000	0.1	100
Nitrate	1	1000	0.1	100
Sulfate	10	1000	1.0	100

<u>Std. 4</u>	<u>Concentration</u> (mg/L)	<u>Standard</u> <u>to be diluted</u> (ppm)	<u>Stock</u> (mLs)	<u>Final</u> <u>Volume</u> (mLs)
Fluoride	10	1000	1	100
Chloride	100	1000	10	100
Bromide	10	1000	1	100
Nitrate	10	1000	1	100
Sulfate	100	1000	10	100

<u>Std. 5</u>	<u>Concentration</u> (mg/L)	<u>Standard</u> <u>to be diluted</u> (ppm)	<u>Stock</u> (mLs)	<u>Final</u> <u>Volume</u> (mLs)
Fluoride	20	1000	2	100
Chloride	200	1000	20	100
Bromide	20	1000	2	100
Nitrate	20	1000	2	100
Sulfate	200	1000	20	100

<u>Std. 6</u>	<u>Concentration</u> (mg/L)	<u>Standard</u> <u>to be diluted</u> (ppm)	<u>Stock</u> (mLs)	<u>Final</u> <u>Volume</u> (mLs)
Fluoride	50	1000	5	100
Chloride	300	1000	30	100
Bromide	50	1000	5	100
Nitrate	50	1000	5	100
Sulfate	500	1000	50	100

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Eluent - 1.7 m M NaHCO_3 (0.286 g/2L)
1.8 m M Na_2CO_3 (0.382 g/2L)

Regenerant 25 m N H_2SO_4
2.8 mL of 50% H_2SO_4
per 2000 mL

Sample Loop -100 uL
-300 uL (O-Phos)

Flow Rate - 2.0 mL/min.

8.2.3 Using injections of 100 uL of each calibration standard, tabulate peak height or area responses against the concentration. The results are used to prepare a calibration curve for each analyte.

8.2.4 The working calibration curve must be verified on each working day, or whenever the anion eluent is changed, and after every 10 samples. If the response or retention time for any analyte varies from the expected values by more than $\pm 10\%$, the test must be repeated, using fresh calibration standards. If the results are still more than $\pm 10\%$, an entire new calibration curve must be prepared for that analyte.

8.2.5 Non-linear response can result when the separator column capacity is exceeded (overloading). Maximum column loading (all anions) should not exceed about 5000 ppm.

8.2.6 Linear calibration curve will be used.

8.3 Analysis

8.3.1 Check system calibration daily and, if required, recalibrate.

8.3.2 Load and inject a fixed amount of well mixed sample. Flush injection loop thoroughly, using each new sample. Use the same size loop for standards and samples. Record the resulting peak size in area or peak height units. An automated constant volume injection system may also be used.

8.3.3 The typical order of analysis shall be: calibration, initial calibration blank (ICB), 2 DCSs, and then samples. A continuing calibration blank (CCB) and continuing calibration

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verification (CCV) must be analyzed after every 10 samples and again at the end of the run.

8.3.4 The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards. Three times the standard deviation of a retention time can be used to calculate a suggested window size for a compound. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

8.3.5 Different dilutions may be required to obtain the concentration of individual anions at the lowest possible reporting limit.

8.3.6 If the resulting chromatogram fails to produce adequate resolution, or if identification of specific anions is questionable, spike the sample with an appropriate amount of standard and reanalyze.

8.4 Conclusion

8.4.1 Shut off the I.C. Clean all glassware, apparatus, and the work area.

8.4.2 Return all samples to sample receiving.

8.4.3 Complete the data package making sure it is filled out properly and all results are highlighted. See the attached example. Be sure to include copies of the special instructions. Submit the package to a peer for data review for approval.

9. QA/QC Requirements

9.1 Before performing any analyses, the analyst should demonstrate the ability to generate acceptable accuracy and precision with this method, using a laboratory control standard and the procedure outlined below.

9.1.1 Into a minimum of four 100 mL volumetric flasks, prepare separate dilutions of the stock DCS solutions as described in Section 7.7.

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- 9.1.2 Analyze the four aliquots according to the procedure in Section 8.3.
- 9.1.3 Calculate the average percent recovery (R) and the standard deviations of the percent recovery (s) for the results.
- 9.1.4 Calculate upper and lower control limits for method performance using the R and s calculated in Section 9.1.3 above as follows:

$$\text{Upper Control Limit (UCL)} = R + 3s$$
$$\text{Lower Control Limit (LCL)} = R - 3s$$

- 9.1.5 For DCS spikes in reagent water the results should be comparable, within the control limits, to the following:

<u>Analyte</u>	<u>Mean Recovery</u>	<u>Standard Deviation</u>
Chloride	97.7	0.0047
Fluoride	103.1	0.0009
Nitrate-N	100.9	0.0041
Sulfate	102.1	0.066
O-Phos-P	100.4	0.019

For analytes for which no limits are given control limits should be based on historical data. Control limits for all analytes should be based on historical data when it becomes available.

- 9.1.6 If the data are not comparable within the control limits, review potential problem areas and repeat the test.
- 9.1.7 Method performance results for each analyst should be filed in the QA office.
- 9.2 QC Samples
- 9.2.1 A blank is required with every batch of 20 or less samples.
- 9.2.2 Duplicate laboratory control samples must be analyzed with every batch of 20 or less samples.

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9.2.3 Continuing calibration blank (CCB) and continuing calibration verification (CCV) samples must be analyzed after every tenth sample. A CCV and CCB must also be analyzed at the completion of the day's analyses.

9.2.4 Duplicates may be required for project specific QC.

9.2.5 Spikes may be required for project specific QC.

9.3 Acceptance Criteria

9.3.1 Blanks must be less than two times the reporting limit.

9.3.2 Average DCS requirements are summarized in the following table:

<u>Analyte</u>	<u>Accuracy Control Limits**</u>	
Fluoride	88 - 112	% Recovery
Chloride	92 - 108	% Recovery
Bromide	90 - 110	% Recovery
Nitrate	91 - 109	% Recovery
Sulfate	93 - 107	% Recovery

9.3.3 The requirements for the RPD for the DCS are summarized in the following table:

<u>Analyte</u>	<u>Precision Control Limits**</u>
Fluoride	15% RPD
Chloride	10% RPD
Bromide	20% RPD
Nitrate	10% RPD
Sulfate	15% RPD

**Subject to change in accordance with Enseco QAPP.

9.3.4 For the DCS data to be considered acceptable, 80% of the acceptance criteria in Sections 9.2.2 and 9.2.3 must be achieved if an IC scan is required.

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9.3.5 Standard checks must be within 10% of the expected value.

9.3.6 There are no acceptance criteria for project specific QC.

9.3.7 The calibration curve for each analyte must have a correlation coefficient of 0.995 or greater.

9.4 Corrective Actions

9.4.1 If acceptance criteria described in Section 9.3 are not met, the analysis sequence must be terminated until the source of the problem can be identified and corrected.

9.4.2 If the blank is more than two times the reporting limit, the entire batch of samples must usually be repped/reanalyzed or anomalized.

9.4.3 Follow the corrective actions outlined in the current Enseco QAPP Manual.

10. Calculations

10.1 Prepare separate calibration curves for each anion of interest by plotting peak size in area or peak height units of standards against concentration values.

10.2 Compute sample concentration by comparing sample peak response with the standard curve.

10.3 Calculations are done automatically using the Dionex software.

11. Reporting Requirements

11.1 Units

Report results in mg/L for aqueous samples and mg/kg for soil samples.

11.2 Limits

11.2.1 The reporting limits for the target anions are listed in the following table:

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<u>Analyte</u>	<u>Reporting Limit</u> <u>Water (mg/L)</u>
Fluoride	0.1
Chloride	3
Bromide	0.2
Nitrate	0.1
Sulfate	5

11.2.2 If dilutions were required for any reason the reporting limit is multiplied by the dilution factor.

11.2.3 If the blank shows contamination (but less than two times the reporting limit) the samples will be flagged with a "B".

11.3 Significant Figures

11.3.1 Range Sig. Figures

< 10	2
10	3
> 10	3

11.3.2 Observed values below the detection limit must be reported as not detected (ND).

11.4 Document any non-standard procedure or any anomalies.

12. Review Requirements

12.1 All data packages must be complete. They must be properly signed and dated. Completed data packages consist of: coversheet, analyst checklist, special instructions, instrument calibration, all chromatograms, and benchsheet. The benchsheet must include analyst name, date, parameters, final concentrations of all samples and QC, reporting limits, standard verification information and expiration dates.

12.2 Confirm that the special instructions were appropriately observed.

12.3 Check any calculations.

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12.4 Check the reporting limits. The standard reporting limit must be multiplied by any dilution factor.

12.5 Verify that the results were correctly anomalized.

12.6 Ensure that peaks named in the chromatogram report are the peaks that should be named. Experience in interpreting chromatograms is required to do this.

13. References

13.1 Method Source

This method is based on EPA Test Method 300.0 (Method A) entitled, "The Determination of Inorganic Anions in Water by Ion Chromatography," December 1989.

13.2 Related Documents

13.2.1 Enseco QAPP

13.2.2 M-EQA-0002 Internal QC checks--Laboratory Performance QC

13.2.3 LM-RMA-1074 D.I. Leach

13.3 Deviations from Source Method and Rationale

13.3.1 The columns specified in the referenced method have been substituted. This change was motivated by advances in the technology which occurred after publication of the method.

13.3.2 Although the source method does not recommend fluoride analysis by IC it will be used occasionally by an experienced chromatographer knowledgeable in the particular problems.

13.3.3 Nitrite has been deleted from the list of target anions because it elutes so close to chloride.

13.3.4 Enseco's Quality Assurance Plan will be followed instead of the quality program outlined in Method 300.0, Section 10.

13.4 SOP Updates (Revision 2.0 to Revision 3.0)

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13.4.1 The reporting limits for fluoride and bromide were revised.

13.4.2 Range for chloride was changed to 300 mg/L for chloride.

13.4.3 "Working" standards was changed to "secondary" standards and the need for a secondary standard concentration for chloride and sulfate was deleted.

B3

TOTAL DISSOLVED SOLIDS

STANDARD
OPERATING
PROCEDURE

Subject or Title:

Total Dissolved Solids
(TDS)

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Revision:
4.0

Effective Date:
May 21, 1991

Supersedes: 3.0

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1. Scope and Application

1.1 Analytes

This method is used for the determination of Total Dissolved Solids, also known as Filterable Residue.

1.2 Reporting Limits

The method reporting limit is 10 mg/L.

1.3 Applicable Matrices

This method is applicable to drinking, surface, and saline waters, domestic, and industrial wastes.

Prepared by

Date:

Management Approval:

Date:

QA Officer Approval:

Date:

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1.4 Dynamic Range

This method covers the range from 10 mg/L and up. It is a gravimetric method and there is no upper limit. As a practical matter, the final residue weight should be limited to about 200 mg.

1.5 Analysis Time

The analytical time is about 30 minutes per sample, not including drying time. The total drying time is variable and can be as long as 24 to 48 hours.

2. Method Summary

A well-mixed sample is filtered through a glass fiber filter. The filtrate is evaporated at 103°C and dried to a constant weight at 180°C.

3. Comments

3.1 Interferences

- 3.1.1 Some samples may contain hygroscopic salts which require prolonged drying, desiccation, and rapid weighing.
 - 3.1.2 Samples containing high concentrations of bicarbonate may require careful prolonged drying to ensure that all the bicarbonate is converted to carbonate.
 - 3.1.3 Too much residue in the pans will crust over, entrapping water that will not be driven off during drying. Total residue should be limited to about 200 mg.
 - 3.1.4 Oil and grease in the samples will cause unreliable results due to difficulty in drying to constant weight.
 - 3.1.5 Some samples may have fine suspended solids which pass through the glass fiber filter causing high results.
 - 3.1.6 Aluminum pans should not be used. Components in some samples may react to form aluminum compounds causing unreliable results.
-

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3.2 Helpful Hints

- 3.2.1 Conductance multiplied by a factor between 0.55 and 0.81 gives a rough approximation of TDS. The exact relationship depends on the compounds present in the samples and may not hold for very high concentrations or samples containing nonionic species or samples with conductances greater than 10,000 umho/cm or less than 50 umho/cm.
- 3.2.2 The filtrate from the TSS procedure (LM-RMA-1062) may be used for TDS determination.

4. Safety Issues

Normal laboratory safety procedures should be followed. There are no special concerns with this method.

5. Sample Collection, Preservation, Containers and Holding Times

- 5.1 Samples are to be collected in plastic bottles. A minimum of 100 mL is required and may or may not be filtered in the field.
- 5.2 Refrigerate all samples to 4°C.
- 5.3 The holding time for TDS is 7 days.

6. Apparatus

- 6.1 Glass fiber filter disks, 47 mm (Gelman Type A/E). The filters should be rinsed with deionized water and dried before use. Assemble the filtration apparatus, turn on the vacuum pump, and place a filter on the holder. Rinse the filter with three 20 mL portions of deionized water, dry at 103°C and store in a desiccator.
- 6.2 Vacuum filtration apparatus.
- 6.3 Vacuum pump equipped with moisture trap.
- 6.4 Porcelain evaporating dishes. These must be thoroughly cleaned, rinsed with deionized water, and baked at 180°C for at least one hour before use. Store in a desiccator.

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- 6.5 Desiccators providing space for storage of samples in process separate from filters and evaporating dishes.
- 6.6 Drying ovens set at 103°C and 180°C. Separate ovens should be maintained at the appropriate temperatures if possible.
- 6.7 Steam bath
- 6.8 Thermometers, NBS traceable.
- 6.9 Analytical balance capable of measurements to 0.0001 g.
- 6.10 Conductivity meter and associated apparatus.
- 6.11 Tweezers, graduated cylinders, and other general laboratory apparatus. Glassware must be thoroughly washed with detergent and rinsed with deionized water.

7. Reagents and Standards

- 7.1 Deionized water (ASTM Type II or equivalent)
 - 7.2 All reagents and standards required for measurement of conductance (see SOP LM-RMA-1062).
 - 7.3 DCS Solution
- Use an ERA Minerals Sample or a sample from another reliable source.

8. Procedure**8.1 Preparation**

- 8.1.1 Check samples for special instructions, making copies to include with the data package. Obtain samples from sample receiving. Fill out the benchsheet. See example benchsheet.
- 8.1.2 Measure and record the conductance of each sample before analysis. An accurate value, corrected for temperature and cell constant, must be obtained. If the conductance has already been measured, this value may be used. Check the Short Holding Time Sheets; TDS has been placed on them in order to identify all samples requiring conductivity. **THE CONDUCTANCE MUST BE RECORDED ON THE BENCH SHEET FOR EACH SAMPLE.**

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- 8.1.3 If samples are visibly oily, this should be noted on the benchsheet.
- 8.1.4 Mark the evaporating dishes with appropriate identification. Weigh dishes for each sample to the nearest 0.0001 g. Only use dishes which have previously been baked at 180°C and stored in a desiccator.
- 8.1.5 Determine the proper volume of each sample to be analyzed.
- 8.1.5.1 If the sample has a conductance less than 3,000 umho/cm, 100 mL should be used.
- 8.1.5.2 If the conductance is 3,000 to 10,000 umho/cm, use 20 mL of sample. Dilute volumes of <100 mL to 100 mL final volume with deionized water.
- 8.1.5.3 If the conductance is extremely high (more than about 10,000 umho/cm), smaller volumes may be used. Consult your supervisor.
- 8.1.5.4 If there is limited sample volume, a smaller amount of sample must be analyzed. This must be noted on an anomaly sheet.

8.2 Analysis

- 8.2.1 Thoroughly rinse the entire filtration apparatus with deionized water before filtering each sample. Assemble the filtration apparatus and turn on the vacuum pump. Only use filters which have been previously rinsed, dried, and stored in a desiccator. The patterned side of the filter must face down.
- 8.2.3 Shake the sample vigorously, measure the required volume into a graduated cylinder, and rapidly transfer to the filtration apparatus. All samples are to be filtered, whether or not they have been filtered in the field.

NOTE: If the sample contain large amounts of insoluble material, a portion may be poured off after mixing and allowed to settle or an aliquot maybe taken and centrifuged, before filtering. Do not pour directly

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from the bottle without mixing. This will alter the sample composition and adversely affect subsequent analyses.

- 8.2.4 After the sample has been filtered, rinse the graduated cylinder into the filtration apparatus with three 10 mL portions of deionized water. Continue vacuum for three minutes after filtration is complete.
- 8.2.5 Transfer the filtered sample to the preweighed evaporating dish. Rinse the receiving flask into the dish to ensure complete transfer of the sample.
- 8.2.6 Place the samples in the drying oven set at 103°C and evaporate to dryness. Record the date and time that the samples were placed in the oven and the temperature of the oven on the benchsheet. Alternately, the samples may be placed on a steam bath and evaporated to dryness.
- 8.2.7 Prepare new filters and dishes to replace the ones you have used. Instructions are given in Sections 6.1 and 6.4 respectively.
- 8.2.8 When the samples are dry, transfer to the drying oven set at 180 +/- 2°C and heat for a minimum of one hour. If a number of samples are being dried simultaneously, additional time may be necessary. Record the date and time that the samples are placed in the drying oven. In addition, record the oven temperature on the bench sheet at the beginning and end of the drying period.
- 8.2.9 Cool the samples in a desiccator for two to three hours. Remove and weigh one tray crucibles at a time to prevent absorption of moisture. Record the date and time of this measurement on the bench sheet.
- 8.2.10 Return the samples to the oven and bake for another hour. Cool the samples in a desiccator and weigh. Record the date, time, and oven temperatures as above. Repeat this cycle again if the weight changes by more than 1.0 mg. If a constant weight is not achieved within three drying cycles, note this on an anomaly sheet. Do not continue the drying, cooling, weighing cycle beyond three times.

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8.3 Conclusion

- 8.3.1 Clean all glassware, apparatus and the work area.
- 8.3.2 Return all samples to sample receiving.
- 8.3.3 Calculate the results following the calculation in section 10. Circle the lowest final weight of the two weights that varied between 1.0 mg. This is used as the final weight for calculating TDS. NOTE: If a constant weight is not achieved within three drying cycles, circle the lowest final weight. This is used as the final weight for calculating TDS.
- 8.3.4 Complete the data package making sure it is properly filled out and the results are highlighted. See sample example. Be sure to include copies of the special instructions.

9. QA/QC Requirements

9.1 QC Samples

- 9.1.1 A blank is required with every batch of 20 or less samples. The blank is prepared using 100 mL of deionized water. It is to be taken through all steps of the analysis, including filtration. It is not necessary to measure the conductance.
- 9.1.2 A DCS pair is required with each batch of 20 or less samples. These are to be taken through all steps of the analysis, including filtration. It is not necessary to measure their conductances. DCS are prepared using 100 mLs of standard.
- 9.1.3 Duplicates may be required for project specific QC.
- 9.1.4 Spikes may be required for project specific QC. Spikes may be analyzed by mixing equal volumes of DCS and sample. Calculation of the result should be based on the actual volume of sample used in the spike (one half the total volume). The "spiking concentration" is equal to the DCS value.

9.2 Acceptance Criteria

- 9.2.1 Weight change for blanks must be between -0.5 and 2 mg.

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9.2.2 Average DCS recovery must be 90 - 110%.

9.2.3 The RPD for the DCS must be less than 10%.

9.2.5 There are no acceptance criteria for project specific QC.

9.3 Corrective Action Required

9.3.1 Follow the corrective actions outlined in the current Enseco QAPP Manual.

9.3.2 Poor results for the DCS's is usually due to contamination. Be sure all glassware and the filtration apparatus is clean and rerun the samples.

9.3.3 High blanks are also the result of contamination. Low blanks indicate that the evaporating dishes were not completely dry when the initial weight was measured. Rerun the samples.

9.3.4 If the TDS/COND ratio falls outside the limits (0.55-0.81) the sample should probably be rerun for conductance and then for TDS. Consult your supervisor.

10. Calculations

10.1 Calculate blank residue weight:

$$\text{BLANK} = (W2 - W1) \times 1000$$

BLANK = Blank residue weight, mg

W2 = Final blank weight, g

W1 = Initial blank weight, g

10.2 Calculate samples and DCS's:

$$\text{RESIDUE} = (W2 - W1) \times 1000$$

$$\text{TDS} = (\text{RESIDUE}) \times 1000 / \text{VOL}$$

RESIDUE = Sample residue weight, mg

W2 = Final weight, g

W1 = Initial weight, g

TDS = TDS result, mg/L

VOL = Sample Volume, mL

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10.3 Calculate the reporting limit

$$\text{Rep. Limit (mg/L)} = \text{Residue (mg)} \times 1000 / \text{Vol. (mL)}$$

This is to be done only if the blank is greater than 1 mg or if a sample volume less than 100 mLs was used. Otherwise, the reporting limit is 10 mg/L.

If the blank is between 1 and 2 mg, the reporting limited is based on the blank residue weight. In all other cases, use 1 mg.

The reporting limit is based on either 100 mL OR the actual volume used, whichever is lower.

10.4 Calculate the TDS/COND ratio.

$$\text{TDS/COND ratio} = \text{Measured TDS} / \text{Measured Conductivity}$$

11. Reporting Requirements

11.1 Units

Report all results in mg/L

11.2 Limits

Results below the reporting limit are reported as ND.

11.3 Significant Figures

Significant figures to report are:

<u>Range</u>	<u>Significant Figures</u>
<10	2
≥10	3

DCS's are reported to three significant figures

11.4 LIMS Data Entry

Since drying times will vary, the analysis date reported will be the date on which the samples were filtered. The holding time is calculated from this analysis date.

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11.5 Anomalies

Document any non-standard procedure or any anomalies. Some common anomalies are:

1. Insufficient sample. Reporting limits changed accordingly.
2. Filtrate is turbid.
3. TDS/COND ratio is outside the limits. TDS & COND were rerun and confirmed. The sample contains high concentration of TDS or may contain nonionic species. The conductivity is greater than 10,000 or less than 50 umho/cm so the rule does not apply.
4. Holding time missed.
5. Samples are visibly oily.

See the attached example benchsheet

12. Review Requirements

- 12.1 All data packages must be complete. They must be properly signed and dated. See example benchsheet.
- 12.2 Confirm that the special instructions were appropriately observed.
- 12.3 Check all sample calculations.
- 12.4 Check the reporting limits.
- 12.5 Verify that all anomalies are documented.
- 12.6 The TDS/COND ratio should fall between 0.55 - 0.81. This ratio may not hold for nonionic species or samples high in TDS. It also may not hold for samples with conductances greater than 10,000 or less than 50 umho/cm. Anomalize results that do not fall within these limits.

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13. References

13.1 Source Method: EPA Method 160.1

13.2 Related Documents

13.2.1 Standard Methods "Total Dissolved Solids Dried at 180°C", 17th Edition, 1989.

13.2.2 Enseco QAPP

13.2.3 M-EQA-0002 Internal QC Checks--Laboratory Performance QC

13.2.4 LM-RMA-1071: pH, Specific Conductance, and Alkalinity:
Automatic Titrator

13.2.5 LM-RMA-1062: Total Suspended Solids (TSS)

13.3 Deviations from Source Method and Rationale

13.3.1 A 103° oven may be used for the evaporation of the sample to dryness.

13.3.2 Constant weight, as defined by the Inorganic Technical Committee of Enseco, is a weight change of 1.0 mg between weighings.

13.3.3 Samples will not be taken through the drying, cooling, weighing cycle more than three times. This is a policy approved by the Enseco Inorganic Technical Committee.

13.4 Updates to SOP (Revision 3.0 to Revision 4.0)

13.4.1 Provision for evaporation of samples on steam bath included.

13.4.2 Maximum number of drying cycles stipulated.

13.4.3 Constant weight defined as weight change of 1.0 mg.

B4

AMMONIA NITROGEN

Subject or Title:		Page <u>1</u> of <u>9</u>
Nitrogen, Ammonia (Technicon AAI)		
SOP No.: LM-RMA-1114	Revision No.: Original	Effective Date: January 23, 1991
Supersedes: N/A		

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1. Scope and Application

1.1 Analytes

This method is applicable to the determination of ammonia as nitrogen.

1.2 Applicable Matrices

1.2.1 This method is applicable to the analysis of surface and saline waters, and domestic and industrial wastes. NPDES samples must be distilled before analysis. (See SOP LM-RMA-1085).

Prepared by:	Date:
Jennifer Franzen	January 23, 1991
Management Approval:	Date:
<i>[Signature]</i>	1/24/91
QA Officer Approval:	Date:
<i>[Signature]</i>	1/24/91

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1.2.2 This method is also applicable to the analysis of soils and wastes following a DI leach (SOP LM-RMA-1074).

1.2.3 Dilutions may be made for high concentrations.

1.3 Reporting Limits

The Enseco reporting limit for ammonia is 0.10 mg/L for waters and 0.50 mg/kg for soils and wastes.

1.4 Dynamic Range

The method covers the range from 0.1 mg/L to 3.0 mg/L in waters, soils and wastes. The range may be extended by making dilutions for higher concentrations.

1.5 Analysis Time

The approximate analysis time is 2 hours for a batch of 20 samples.

2. Method Summary

Samples are analyzed on the Technicon Autoanalyzer II which performs the automated colorimetric analysis.

Ammonia is analyzed using the Berthelot Reaction, forming a blue compound with sodium phenoxide and sodium hypochlorite. The color is intensified by the addition of sodium nitroprusside. EDTA prevents the precipitation of calcium and magnesium hydroxides.

3. Comments

This method is extremely sensitive to changes in pH.

3.1 Interferences

3.1.1 Calcium and magnesium may precipitate and clog the ammonia channel. EDTA is used to prevent this. The EDTA may not be effective on salt water samples.

3.1.2 Color and turbidity in the samples will interfere. They may be removed by filtration. Samples with color interferences must be diluted until no interference is detected.

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3.1.3 The samples, standards, and rinse water must all contain the same concentration of acid.

3.2 Helpful Hints

3.2.1 Samples, standards, and rinse water work best if the concentration of the acid is 0.2% sulfuric acid.

4. Safety Issues

- 4.1 All employees are expected to be familiar with and follow the procedures outlined in the Enseco-RMAL safety plan. Lab coats and safety glasses are required in all laboratory areas at all times. If you have any questions or safety concerns, see your supervisor or safety officer.
- 4.2 Wear gloves and apron when handling concentrated acids, bases and solvents. Transport only in approved carriers. Avoid breathing fumes and vapors; handle in a fume hood. Neutralize and clean up any spills immediately. In case of skin contact, flush affected area with water for at least 15 minutes. Notify your supervisor or safety officer of any spills or exposures.
- 4.3 No solvents of any kind or in any amount are to be disposed of in the sinks. Dispose of all solvents in suitable waste containers.
- 4.4 Wear gloves and apron when performing extractions. Extractions are to be performed in a fume hood.
- 4.5 Wear gloves, apron, and face shield when performing digestions or distillations. Digestions and distillations are to be performed in a fume hood.
- 4.6 Samples and other solutions containing high concentrations of toxic materials must not be flushed down the sinks, but are to be disposed of in suitable waste containers.
- 4.7 All samples are to be considered hazardous. Wear gloves and handle in a fume hood as much as possible.
- 4.8 Phenol is highly toxic and can be absorbed through the skin or by inhalation of the vapor. Wear gloves and handle in the fume hood.

Nitrogen, Ammonia (Technicon AAI)

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January 23, 1991**5. Sample Collection, Preservation and Holding Times**

- 5.1 Samples are to be collected in glass bottles whenever possible.
- 5.2 Samples are to be preserved by adding sulfuric acid (2 mL per 1000 mL sample) and refrigerated at 4°C.
- 5.3 The holding time for ammonia in water is 28 days. There is no holding time for soils or wastes.

6. Apparatus

- 6.1 The technicon AAI equipped with the proportioning pump, autosampler, and colorimeter with 660-nm filters and 50-mm flow cell.
- 6.2 Miscellaneous laboratory apparatus and glassware.

7. Reagents and Standards

- 7.1 ASTM Type II Water (Milli-Q or equivalent).
- 7.2 Sulfuric acid 0.2%

Slowly add 4 mL of concentrated sulfuric to 2 liters of ASTM type water. This will be used to make standards and any dilutions that may be required.

- 7.3 Sodium phenolate

Add 20.75 g of phenol (crystals) to 100 mL of distilled water in a 250 mL volumetric. Do this in the hood. Allow the phenol to dissolve some then add 8.0 g of sodium hydroxide pellets. Cool to room temperature and bring to volume. Store in an amber glass container and prepare fresh every two weeks.

- 7.4 Sodium Hypochlorite Solution

Dilute 100 mL of reagent grade Hypochlorite (5%) to 200 mL with distilled water. Commercial bleach (5%) may be used in the same manner.

- 7.5 Sodium Nitroprusside Solution (0.05%)

Dissolve 0.125 g of sodium nitroprusside in 250 mL of deionized water. Store in an amber container and prepare fresh monthly.

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7.6 Disodium EDTA

Add 50 g of sodium EDTA to 800 mL of deionized water. Use a pH electrode to adjust the pH to 8.0 by slow addition of 50% sulfuric acid. Cool to room temperature and bring to volume. Add 1 mL of Brij-35 and mix.

7.7 Ammonia Stock Standard

Dry Ammonium Chloride at 105°C. Dissolve 3.819 g in 600 mL of deionized water, add 2 mL of concentrated sulfuric acid, and dilute to 1000 mL with deionized water. This solution is to be obtained from standards prep.

7.8 Intermediate Standard

Pipette 1.0 mL of stock standard into a 100 mL volumetric flask and dilute to volume with 0.2% sulfuric acid.

7.9 Working Standards

Prepare dilutions as indicated below. All dilutions should be made with 0.2% sulfuric acid.

Volume Intermediate Standard	Final Volume	Concentration mg/L as N
1.0 mL	100 mL	0.1
2.0 mL	100 mL	0.2
5.0 mL	100 mL	0.5
10.0 mL	100 mL	1.0
20.0 mL	100 mL	2.0
30.0 mL	100 mL	3.0

7.10 DCS Solution

Use ERA nutrients for DCS solutions. These must be preserved properly with sulfuric acid to 0.2%.

8. Procedure

8.1 Sample Preparation

8.1.1 Check the pH of all samples to ensure that they have been properly preserved. If necessary, add sulfuric acid to produce a final concentration of 0.2% and indicate on the benchsheet.

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8.1.2 Soils and wastes must be taken through the D.I. Leach procedure before analysis. (LM-RMA-1074)

8.1.3 NPDES samples for ammonia must be distilled before analysis (LM-RMA-1085).

8.2 Instrument Set-up and Calibration

8.2.1 Turn on the colorimeter and allow 20 min. warmup time.

8.2.2 Set up the proportioning pump for pumping reagents. Pump all reagents except Nitroprusside. The pH needs to adjust before pumping this reagent. Pump the Nitroprusside after the other reagents have been pumping for five minutes.

8.2.3 Turn on the chart recorder and allow the baseline to stabilize.

8.3 Analysis

8.3.1 Standards are to be analyzed in order of decreasing concentration. A linear fit should be used.

8.3.2 Samples exceeding the high standard require a dilution. Samples below 0.1 mg/L are reported as ND.

9. QA/QC Requirements

9.1 QC Samples

9.1.1 A blank (0.2% sulfuric acid) is required with every batch of 20 or less samples.

9.1.2 Two DCS samples are required for each batch of 20 or less samples. Use ERA nutrients.

9.1.3 Duplicates may be required for project specific QC.

9.1.4 Spikes may be required for project specific QC. Dilute 1 mL Intermediate Standard to 10 mL with sample. The spiking concentration is 1 mg/L.

9.2 Acceptance Criteria

9.2.1 Blanks must be less than two times the reporting limit.

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9.2.2 Average DCS recovery must be 93-107% .

9.2.3 The RPD for the DCS must be less than 10%

9.2.4 There are no acceptance criteria for project specific QC.

9.2.5 Standard checks must be within 10% of the expected value.

9.2.6 The calibration curve must have a correlation coefficient of 0.995 or greater.

9.3 Corrective Actions

9.3.1 Follow the corrective actions outlined in the current Enseco QAPP Manual.

9.3.2 If the blank is more than two times the reporting limit, the entire batch of samples must usually be reprepped and reanalyzed. Consult your supervisor.

9.3.3 Check for contamination in reagents and standards. Be sure the reagents were made correctly and have not exceeded their expiration dates.

9.3.4 All samples and standards must contain 0.2% sulfuric acid.

10. Calculations

10.1 Measure and record the peak height for each sample.

10.2 Prepare a calibration curve from the standard data. Use a linear least squares program.

10.3 Calculate the sample concentrations from the calibration curve and multiply by any dilution factors. Be sure to include dilutions made during prep.

11. Reporting Requirements

11.1 Reporting units are mg/L for waters and mg/kg for non-aqueous samples.

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11.2 Limits

11.2.1 If the blank shows contamination (but is less than two times the reporting limit), the reporting limit is raised to the blank value.

11.2.2 If dilutions were required due to insufficient sample, interferences, or other problems, the reporting limit is multiplied by the dilution factor.

11.3 Significant Figures

11.3.1 Range Sig. Figures

< 10	2
10	3
> 10	3

11.3.2 Observed values below the detection limit must be reported as not detected (ND) .

11.4 LIMS Data Entry

The usual standards for data entry apply.

11.5 Anomalies

Document any non-standard procedures or anomalies. Some common anomalies are:

11.5.1 Sample diluted for color interference.

11.5.2 Sample pH was neutral preserved with sulfuric acid before analysis.

12. Review Requirements

12.1 All data packages must be complete. They must be properly signed and dated.

12.2 Confirm that the special instructions were appropriately observed.

12.3 Confirm that there were no unusual peaks by checking the chromatogram.

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12.4 Check calculations.

12.5 Check the reporting limits. The standard reporting limit must be multiplied by any dilution factor or raised to any blank contamination value.

12.6 Verify that the results were correctly anomalized.

13. References

13.1 Source Method: EPA 350.1 Nitrogen, Ammonia (Colorimetric, Automated Phenate)

13.2 Related Documents

13.2.1 4500-NH3.H Automated Phenate Method Standard Methods

13.2.2 Enseco QAPP

13.2.3 M-EQA-0002 Internal QC Checks--Laboratory Performance QC

13.2.4 Enseco SOP LM-RMA-1039 Rev. 1.0 Nitrogen Ammonia 350.1 EPA

13.3 Deviations from Source Method and Rationale

13.3.1 The source method has been expanded to include soils and wastes through the use of a deionized water leach.

13.3.2 The EDTA solution is brought to a specific pH by pH electrode in order to maintain a consistent pH for all analysis.

(Attached are completed example benchsheet and appropriate diagrams or tables.)

Analyst ANALYST Control (ERA 9932 10.6) ^{Example} Date ()
Parameter NH₃ Spike 1.0 mg/L Units mg/L, mg/kg

[illegible]

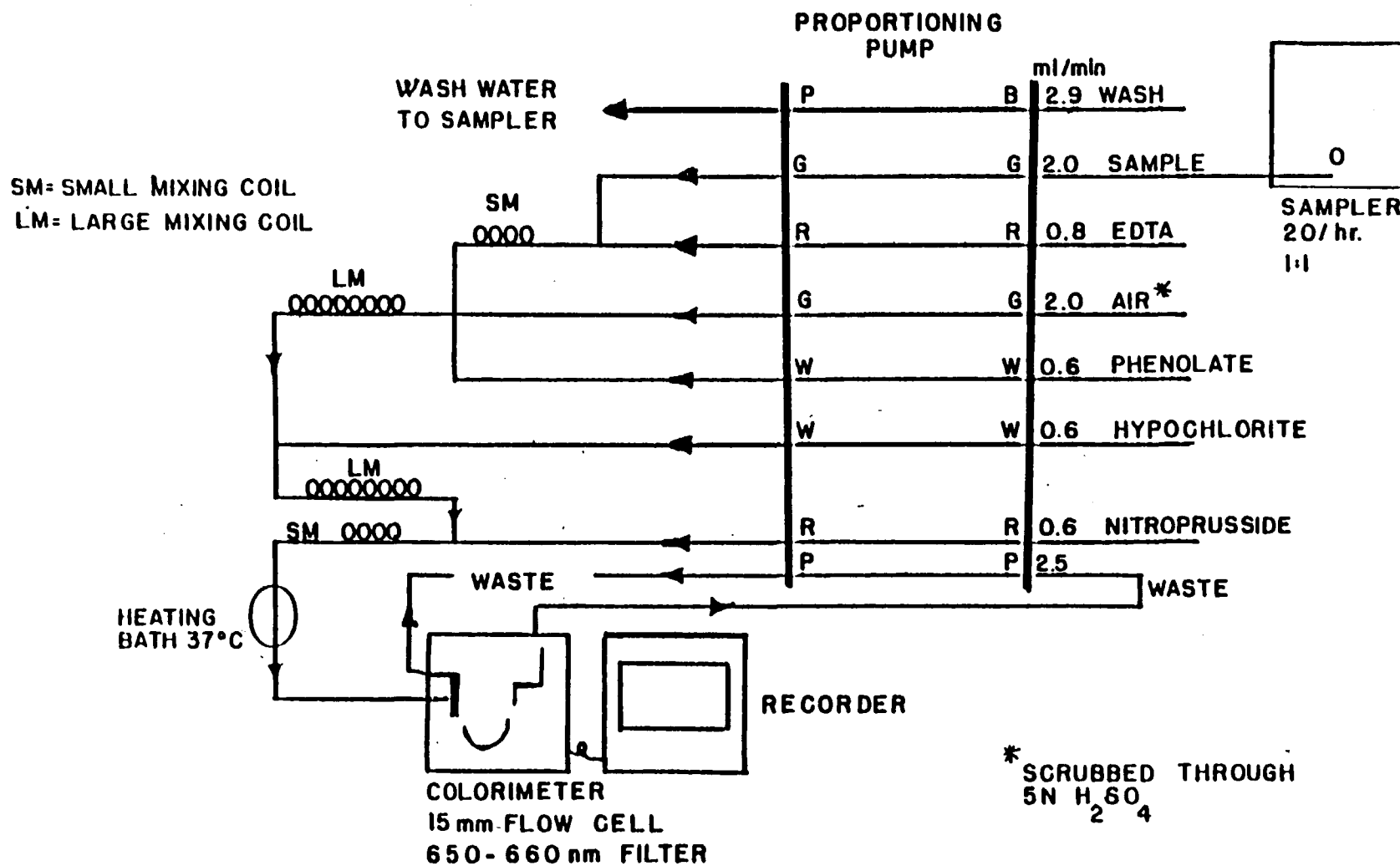


FIGURE 1 AMMONIA MANIFOLD AA I

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NITRATE & NITRITE NITROGEN

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1. Scope and Application

1.1 Analytes

This method is applicable to the analysis of nitrate and nitrite combined. Nitrate can be determined through calculation.

1.2 Applicable Matrices

1.2.1 This method is applicable to the analysis of drinking, surface and saline waters, domestic and industrial wastes.

Prepared by:
Janice Collins

Date:
2/1/93

Technology Manager Approval:

Date:

Management Approval:

Date:

QA Officer Approval:

Date:

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1.2.2 This method is also applicable to the analysis of soils and wastes following a DI leach (See SOP LM-RMA-1074).

1.2.3 Dilutions may be made for high concentrations.

1.3 Reporting Limits

The Enseco Reporting limits for Nitrate + Nitrite is 0.10 mg/L for waters and 0.50 mg/kg for soils and wastes.

1.4 Dynamic Range

The method covers the range from 0.1 mg/L to 3.0 mg/L in waters, 0.5 mg/kg to 15.0 mg/kg in soils and wastes. The range may be extended by making dilutions for higher concentrations.

1.5 Analysis Time

The approximate analysis time is 2 hours for a batch of 20 samples.

2. Method Summary

Samples are analyzed on the Technicon Autoanalyzer II, which performs the automated colorimetric analysis.

Nitrate + Nitrite is analyzed by reducing nitrate to nitrite in a copper - cadmium column. The reduced nitrite plus the nitrite originally present then reacts with Sulfanilamide to form a diazo compound that then couples with N-1-Naphthylethylenediamine dihydrochloride to form a reddish-purple azo dye.

3. Comments

3.1 Interferences

3.1.1 Build up of suspended matter in the reduction column will restrict sample flow. Particulates may be removed by filtration.

3.1.2 High concentrations of iron, copper and other metals will cause low results.

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- 3.1.3 Color and turbidity in the sample will interfere. Turbidity is removed by filtration. Samples with color interference must be diluted until no interference is detected.
- 3.1.4 Samples that contain large concentrations of oil and grease will coat the surface of the cadmium column.
- 3.1.5 Samples preserved with mercuric chloride or sodium thiosulfate must not be analyzed by this method or the reduction column will be degraded.

3.2 Helpful Hints

- 3.2.1 Nitrite may be analyzed alone if the reduction column is bypassed. The carrier must be changed to deionized water, since the samples are unpreserved. Standards must be prepared from a Nitrite stock instead of a Nitrate stock and diluted with deionized water. Nitrite is usually analyzed manually (see Nitrite SOP: LM-RMA-1041).
- 3.2.2 Nitrate is obtained by subtracting the Nitrite result from the Nitrate + Nitrite result.
- 3.2.3 WARNING: Do not let any air bubbles get into the reduction column or it will be ruined. Do not pump deionized water through it; it must be taken off line while buffer is flowing through it, capped and stored filled with buffer.
- 3.2.4 Samples, standards and rinse water work best if concentration of the acid is 0.2% sulfuric acid.

4. Safety Issues

- 4.1 All employees are expected to be familiar with and follow the procedures outlined in the Enseco-Rocky Mountain Analytical Laboratory (RMAL) safety plan. Lab coats and safety glasses are required in all laboratory areas at all times. If you have any questions or safety concerns, see your team manager or safety officer.
- 4.2 Wear gloves and apron when handling concentrated acids, bases, and solvents. Transport only in approved carriers. Avoid breathing fumes and vapors; handle in a fume hood. Neutralize and clean up

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any spills immediately. In case of skin contact, flush affected area with water for at least 15 minutes. Notify your team manager or safety officer of any spills or exposures.

- 4.3 No solvents of any kind or in any amount are to be disposed of in the sinks. Dispose of all solvents in suitable waste containers.
- 4.4 Wear gloves and apron when performing extractions. Extractions are to be performed in a fume hood.
- 4.5 Wear gloves, apron, and face shield when performing digestions or distillations. Digestions and distillations are to be performed in a fume hood.
- 4.6 Samples and other solutions containing high concentrations of toxic materials must not be flushed down the sinks, but are to be disposed of in suitable waste containers.
- 4.7 All samples are to be considered hazardous. Wear gloves and handle in a fume hood as much as possible.
- 4.8 Nitrate + Nitrite waste contains cadmium, which is toxic and carcinogenic. Wear gloves when handling cadmium.

5. Sample Collection, Preservation, and Holding Times

- 5.1 Samples are to be collected in glass bottles whenever possible.
- 5.2 Samples are to be preserved by adding sulfuric acid (2 mL per 1000 mL sample) and refrigerated to 4°C.
- 5.3 The holding time for Nitrate + Nitrite in water is 28 days. There is no holding time for soils or wastes.

6. Apparatus

- 6.1 The technicon AAI equipped with proportioning pump, autosampler, and colorimeter with 520 nm filters and 50 mm flowcell.
- 6.2 Miscellaneous laboratory apparatus and glassware.

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7. Reagents and Standards

7.1 Reagent water (Milli-Q or equivalent) from a polishing system (ASTM Type II or equivalent).

7.2 Sulfuric acid 0.2%

Slowly add 4 mL of concentrated sulfuric acid to 2 liters of reagent water. This will be used to make standards and any dilutions that may be required.

7.3 Color Reagent

Add 100 mL concentrated phosphoric acid, 40 g sulfanilamide and 2 g N-1-Naphthylethylenediamine dihydrochloride to approximately 800 mL of deionized water. Stir until dissolved and dilute to 1 liter with reagent water. Store in brown bottle and keep in dark when not in use. This solution is stable for several months.

7.4 Ammonium chloride - EDTA solution

Dissolve 85 g of reagent grade ammonium chloride and 0.1 g of disodium ethylenediamine tetracetate in 900 mL of deionized water. Adjust the pH to 8.5 with concentrated ammonium hydroxide and dilute to 1 liter. Add 0.5 mL of Brij-35.

7.5 50% Triton X-100 solution

Add 50 mL of Triton X-100 to 50 mL of isopropanol and mix thoroughly.

7.6 2N Hydrochloric Acid

Slowly add 165 mL of concentrated hydrochloric acid to about 600 mL of DI water. Cool to room temperature and dilute to 1 liter with DI water. Add 0.5 mL 50% Triton X-100 and mix.

7.7 Stock Copper Sulfate Solution 'A', 0.01M

Dissolve 2.5 g of copper sulfate in about 600 mL of DI water. Dilute to 1 liter with reagent water and mix.

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7.8 Copper Activating Solution

Dilute 50 mL of stock copper sulfate 'A', 0.01M to 100 mL with DI water. Add 0.1 mL 50% Triton X-100.

7.9 Stock Standard, 1000 mg/L as N

Dissolve 7.218 g dried potassium nitrate in reagent water and dilute to 1000 mL. Add 400 μ L chloroform as a preservative. This solution is stable for 3 months. It is to be obtained from standards prep.

7.10 Intermediate standard, 10 mg/L

Pipette 1.0 mL of stock standard into a 100 mL volumetric flask and dilute to volume with 0.2% sulfuric acid.

7.11 Working Standards

Prepare dilutions as indicated below. All dilutions should be made with 0.2% sulfuric acid.

<u>Volume Intermediate Standard</u>	<u>Final Volume</u>	<u>Concentration mg/L as N</u>
1.0 mL	100 mL	0.1
2.0 mL	100 mL	0.2
5.0 mL	100 mL	0.5
10.0 mL	100 mL	1.0
20.0 mL	100 mL	2.0
30.0 mL	100 mL	3.0

7.12 DCS Solution

Use ERA nutrients for DCS solutions. These must be preserved properly with sulfuric acid to 0.2%.

8. Procedure

8.1 Sample Preparation

8.1.1 Check the pH of all samples to ensure that they have been properly preserved. If necessary, add sulfuric acid to produce a pH of less than 2 and anomalize.

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8.1.2 Soils and wastes must be taken through the D.I. Leach procedure before analysis (LM-RMA-1074).

8.2 Instrument Set-up and Calibration

8.2.1 Set up manifold as shown in figure 1.

8.2.2 Turn on the colorimeter and allow 20 minutes warm-up time.

8.2.3 Turn on the nitrogen until the water in the flask is bubbling. DO NOT allow air to be pumped through the column.

8.2.4 Set up the proportioning pump for pumping reagents. Pump all reagents through before hooking up the column.

8.2.5 Recondition the column if necessary by pumping 2N HCl through the buffer line for one minute, the copper activating solution for two minutes, then 2N HCl again for five minutes. Place the buffer line back into the buffer and allow 15 - 20 minutes equilibration time.

8.2.6 Turn on the chart recorder and allow the baseline to stabilize.

8.3 Analysis

8.3.1 Load and start the autosampler. A few sample cups of 3.0 mg/L standard must be loaded at the beginning of the run. When the peaks for these standards appear, adjust the gain control on the colorimeter to obtain a full scale peak.

8.3.2 Standards are to be analyzed in order of decreasing concentration. A linear fit should be used. Standards should be followed by a blank, two DCSs and samples. A standard check and blank should be analyzed every 10 or fewer samples and again at the end of the run.

8.3.3 Samples exceeding the high standard require a dilution. Samples below 0.1 mg/L are reported as ND.

8.3.4 Following analysis, disconnect the reduction column then pump deionized water through the system. Do not pump water through the column. The column should always be stored in buffer.

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9. QA/QC Requirements

9.1 QC Samples

- 9.1.1 A blank (0.2% sulfuric acid) is required with every batch of 20 or less samples.
- 9.1.2 Two DCS samples are required for each batch of 20 or less samples. Use ERA nutrients. Check Certificate of Analysis supplied by ERA for true values.
- 9.1.3 Duplicates may be required for project specific QC.
- 9.1.4 Spikes may be required for project specific QC. Dilute 1 mL of sample with 1 mL of 3.0 mg/L standard. The spiking concentration is 3.0 mg/L.

9.2 Acceptance Criteria

- 9.2.1 Blanks must be less than two times the reporting limit.
- 9.2.2 Average DCS recovery must be 91-109%.
- 9.2.3 The RPD for the DCS must be less than 10%.
- 9.2.4 There are no acceptance criteria for project specific QC.
- 9.2.5 Standard checks must be within 10% of the expected value.
- 9.2.6 The calibration curve must have a correlation coefficient of 0.995 or greater.

9.3 Corrective Actions

- 9.3.1 Follow the corrective actions outlined in the current Enseco QAPP Manual.
- 9.3.2 Check for contamination in the reagents and standards. Be sure all reagents were made correctly and have not exceeded their expiration dates.
- 9.3.3 All samples and standards must contain 0.2% sulfuric acid.
- 9.3.4 Check system for obvious problems such as leaks or plugs.

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9.3.5 Some samples may deactivate the reduction column. Analyze a 2 mg/L Nitrite-N standard and compare to a 2 mg/L Nitrate-N standard. If the results differ by more than 10%, the column needs to be reactivated (see 8.2.5). This problem will also be evident with a sudden drop in standard check recovery.

10. Calculations

- 10.1 Measure and record the peak height for each sample.
- 10.2 Prepare a calibration curve from the standard data. Use a linear least squares program.
- 10.3 Calculate the sample concentrations from the calibration curve and multiply by any dilution factors.
- 10.4 If nitrate is required, subtract nitrite (LM-RMA-1041) results from nitrate + nitrite value.

11. Reporting Requirements

- 11.1 Reporting units are mg/L for waters and mg/kg for non-aqueous samples.
- 11.2 Limits
 - 11.2.1 If the blank shows contamination (but is less than two times the reporting limits), the samples will be flagged with a "B."
 - 11.2.2 If dilutions were required due to insufficient sample, interferences, or other problems, the reporting limit is multiplied by the dilution factor.

11.3 Significant Figures

Any values less than 10 will be reported with 2 significant figures. Values greater than or equal to 10 will be reported with three significant figures.

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11.4 Anomalies

Document any non-standard procedures or anomalies. Some common anomalies are:

11.4.1 Sample diluted for color interference.

11.4.2 Sample pH was neutral. Preserved with sulfuric acid before analysis.

12. Review Requirements

12.1 All data packages must be complete. They must be properly signed and dated. Completed data packages consist of: coversheet, analyst checklist, special instructions, instrument calibration, chromatogram and completed benchsheet. Standard verification information and expiration dates must also be included.

12.2 Confirm that the special instructions were appropriately observed.

12.3 Confirm that there were no unusual peaks by checking the chromatogram.

12.4 Check calculations.

12.5 Check the reporting limits. The standard reporting limit must be multiplied by any dilution factor.

12.6 Verify that the results were correctly anomalized, if necessary.

13. References

13.1 Source Method: EPA 353.2 Nitrogen, Nitrate-Nitrite (Colorimetric, Automated, Cadmium Reduction).

13.2 Related Documents

13.2.1 Enseco QAPP

13.2.2 M-EQA-0002 Internal QC Checks-- Laboratory Performance QC

13.2.3 Enseco SOP LM-RMA-1105, Nitrate + Nitrite; Nitrate (LACHAT Autoanalyses)

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13.3 Deviations from Source Method and Rationale

13.3.1 A purchased reduction column is used. Activating procedure is from technicon Industrial Method No. 824-87T.

13.3.2 The source method has been expanded to include soils and wastes through the use of a deionized water leach.

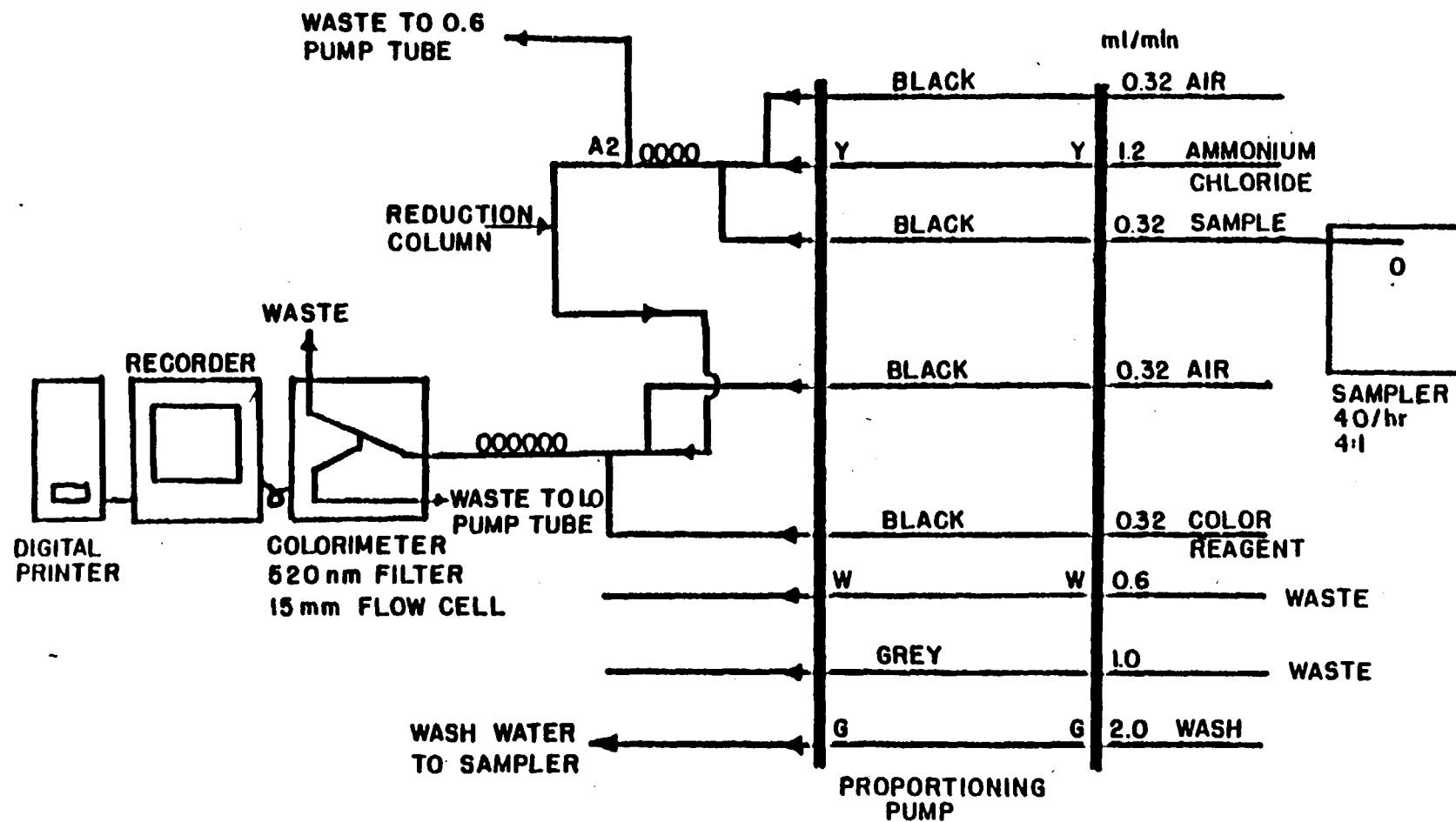


FIGURE 1. NITRATE-NITRITE MANIFOLD AA II

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TOTAL PHENOLICS

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Subject or Title:

Total Recoverable Phenolics
(Automated)Page 1 of 10

SOP No.: LM-RMA-1099

Revision No.:
OriginalEffective Date:
March 13, 1989

Supersedes: None

1. Scope and Application

- 1.1 This method measures steam-distillable phenolic materials which react with the color reagents under the conditions of the analysis.
- 1.2 The detection limit is 0.01 mg/L as Phenol (1 mg/kg for nonaqueous matrices).
- 1.3 This method is applicable to the analysis of drinking, surface and saline waters, domestic and industrial wastes, and soil samples.
- 1.4 The range extends from 0.01 mg/L to 0.5 mg/L (1 mg/kg to 40 mg/kg). The range can be extended by dilution of the samples.
- 1.5 Approximate preparation time is 2 hours for a group of 10 samples. Analytical time is about 20 samples per hour.

2. Summary of Method

The sample is acidified and distilled to separate phenolics from interfering compounds. Phenolics in the distillate react with 4-aminoantipyrine in the presence of potassium ferricyanide at pH 10 to form a reddish-brown dye, which is measured colorimetrically at 505 nm. This is the method commonly used at RMAL. A detection limit of 0.005 mg/L (1 mg/kg for nonaqueous matrices) can be achieved using SOP: LM-RMA-1048.

3. Comments

3.1 Interferences

Prepared by:
Lindsay BreyerDate:
February 27, 1989

Management Approval:

W. Pratt

Date:

3/14/89

QA Officer Approval:

Jan Redenberger

Date:

3/15/89

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3.1.1 Most direct interferences are eliminated by distillation of an acidified sample. Phenolic compounds distill with the water but interfering compounds do not.

3.1.2 Some phenolic compounds are not steam-distillable and will not be determined.

3.1.3 The colors produced by various phenolic compounds are not the same, so the response will depend on the compounds actually present in the samples. Phenol has been selected as the calibration standard since it is not possible to reproduce the mixture of compounds present in the samples. The result obtained will represent the minimum concentration of phenolics present in the sample.

3.1.4 Interference from sulfur compounds is eliminated by acidification and addition of copper sulfate.

3.1.5 Oxidizing agents such as chlorine will oxidize phenolic compounds and must be removed.

3.1.6 Oil may distill over and interfere with the analysis.

3.1.7 Aromatic amines may react with nitrite (if present) to produce phenolic compounds.

3.2 Helpful Hints
None.

4. Safety Issues

4.1 All employees are expected to be familiar with and follow the procedures outlined in the Enseco/RMAL safety plan. Lab coats and safety glasses are required in all laboratory areas at all times. If you have any questions or safety concerns, see your supervisor or safety officer.

4.2 Wear gloves and apron when handling concentrated acids, bases and solvents. Transport only in approved carriers. Avoid breathing fumes and vapors; handle in a fume hood. Neutralize and clean up any spill immediately. In case of skin contact, flush affected area with water for at least 15 minutes. Notify your supervisor or safety officer of any spills or exposures.

4.3 Wear gloves, apron and face shield when performing distillations. Distillations are to be performed under the slot hood.

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4.4 Phenol is extremely toxic and can be absorbed through the skin. Handle only in a fume hood and wear gloves. In case of skin contact, flush with water for at least 15 minutes. Notify your supervisor or safety officer of any exposures.

4.5 Samples, reagents and other solutions containing high concentrations of toxic materials must not be flushed down the sinks, but are to be disposed of in suitable waste containers.

5. Samples Collection and Preservation

5.1 Samples are to be collected in glass containers and preserved by adding sulfuric acid to $\text{pH} < 2$ and refrigerating at 4°C .

5.2 The holding time is 28 days.

6. Apparatus

6.1 All-glass distillation apparatus consisting of 500 mL round-bottom flask with side arm, coil condenser, heating mantle with controller and associated adapters and hardware.

6.2 Recirculating chiller.

6.3 pH meter and electrode.

6.4 Technicon AutoAnalyzer II with sampler, pump, colorimeter, recorder and manifold shown in the attached diagram. Use 50 mm flow cells and 505 nm filters.

6.5 Miscellaneous laboratory apparatus and glassware.

7. Reagents and Standards

7.1 Sulfuric Acid, 50%

Slowly add 50 mL concentrated sulfuric acid to 500 mL deionized water with constant mixing and cool. The reaction is very exothermic and should be done with extreme caution.

7.2 Boiling stones.

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7.3 Copper Sulfate, 10%

Dissolve 100 g cupric sulfate 5-hydrate in deionized water and dilute to 1000 mL.

7.4 Ferrous Ammonium Sulfate Solution

Add 1 mL concentrated sulfuric acid to 500 mL deionized water. Add 1.1 g ferrous ammonium sulfate, mix until dissolved, and dilute to 1000 mL.

7.5 Sodium Hydroxide, 1N

Dissolve 40 g sodium hydroxide in deionized water, cool, and dilute to 1000 mL with deionized water. Store in a plastic bottle.

7.6 Buffered Potassium Ferricyanide

Dissolve 2.0 g potassium ferricyanide, 3.1 g boric acid and 3.75 g potassium chloride in 800 mL deionized water. Adjust the pH to 10.3 with 1 N sodium hydroxide and dilute to 1000 mL. Add 0.5 mL Brij-35 and mix. Prepare weekly.

7.7 4-Aminoantipyrene

Dissolve 0.65 g 4-aminoantipyrene in deionized water and dilute to 1000 mL.

7.8 Phenol Stock Standard, 1000 mg/L

Dissolve 1.000 g phenol in deionized water and dilute to 1000 mL.

7.9 Phenol Intermediate Standard, 10 mg/L

Dilute 1.0 mL 1000 mg/L Stock Standard to 100 mL with deionized water.

7.10 Working Standards

Dilute the 10 mg/L Intermediate Standard with deionized water as follows:

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Aliquot (mL)	Final Vol. (mL)	Conc. (mg/L)
0	100	Blank
1.0	100	0.1
2.0	100	0.2
3.0	100	0.3
4.0	100	0.4
5.0	100	0.5

NOTE: The standards are not distilled with the samples.

7.11 pH test strips

7.12 Starch/iodide test strips

7.13 Lead Acetate test strips

8. Procedure

8.1 Sample Preparation

8.1.1 Measure and record the pH of all water samples. pH test strips may be used.

8.1.2 Check for residual chlorine with starch/iodide test strips. A blue to black color indicates a positive test. Record the result on the bench sheet.

8.1.3 Check for sulfide using lead acetate test strips. A dark color indicates the presence of sulfide. Record the result on the bench sheet.

8.1.4 Measure 200 mL sample into a distillation flask and add a few boiling stones. For soil and waste samples, use 2.0 g and add 200 mL deionized water. Be sure to adjust the pH of soil and waste samples before distillation. Record the exact weight on the bench sheet.

8.1.5 If the chlorine test was positive, add ferrous ammonium sulfate solution until a negative test is obtained.

8.1.6 If the pH is not < 2 , add 50 % sulfuric acid dropwise until it is..

8.1.7 If the sulfide test was positive, add 2 mL 10% copper sulfate.

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- 8.1.8 Assemble the distillation apparatus, turn on the cooling water and hood, and start the distillation. Capture the distillate in a 250 mL beaker.
- 8.1.9 When 150 to 175 mL distillate has been collected, turn off the heating mantle and allow to cool.
- 8.1.10 Add 25 to 30 mL deionized water and resume distillation until 200 mL has been collected. Turn off the heating mantle and clean out the flask when cool. Do not overdistill the samples as this will lead to interferences in the analysis.
- 8.1.11 If the distillate is cloudy, filter through a prerinsed membrane filter.
- 8.1.12 Transfer the distillates to 250 mL glass bottles with teflon caps and refrigerate until they are analyzed.
- 8.1.13 At the end of the day, turn off the chiller.

8.2 Analysis

- 8.2.1 Set up the manifold shown in the attached diagram (Figure 1). Allow the colorimeter and recorder to warm up for at least 30 minutes while pumping reagents (pump deionized water through the sample line).
- 8.2.2 Continue pumping reagents until a steady baseline is obtained. If new pump tubing has been installed, up to 2 hours may be necessary. Adjust the baseline to the bottom of the charts with the baseline control.
- 8.2.3 Fill three cups with the 0.5 mg/L standard, place on the sampler and start. Use the standard cal control to adjust the peaks to full scale. Readjust the baseline if necessary.
- 8.2.4 Place the standards on the sampler in order of decreasing concentration. Follow these with the prep blank, the DCSs and the samples.
- 8.2.5 Start the sampler. Dilute and reanalyze any samples reading off-scale. Be sure to analyze a blank and standard check after every 10 or less samples and again at the end of the run.

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8.2.6 At the end of the day, flush the system thoroughly with deionized water and turn off the instrument.

9. QA/QC Requirements

9.1 QC Samples

- 9.1.1 A blank (deionized water) is required with every batch of 20 or less samples. The blank must be taken through the entire prep and analysis with the samples.
- 9.1.2 Two DCS samples are required with each batch of 20 or less samples. Use an ERA cyanide/phenol sample. These must be taken through the entire prep and analysis with the samples.
- 9.1.3 A blank and standard check are required after every 10 or less samples.
- 9.1.4 Duplicates may be required for project specific QC.
- 9.1.5 Spikes may be required for project specific QC. Add 5.0 mL of the 10 mg/L intermediate standard to 200 mL sample and distill. The spiking concentration is 0.25 mg/L (25 mg/kg).

9.2 Acceptance Criteria

- 9.2.1 If the blank reads greater than 3 times the detection limit, consult your supervisor.
- 9.2.2 DCS recovery must be 88 - 122%.
- 9.2.3 The RPD for DCS samples must be less than 20%.
- 9.2.4 Standard checks must be within 20% of the expected value.
- 9.2.5 There are no acceptance criteria for project specific QC.
- 9.2.6 The calibration curve must have a correlation coefficient of at least 0.995.

9.3 Corrective Action Required

- 9.3.1 Negative peaks usually indicate that the sample pH is too low. Add 1 N sodium hydroxide dropwise to adjust the pH to between 5 and 6.

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9.3.2 Follow the corrective action procedures outlined in the current Enseco QA Plan. If problems persist, consult your supervisor.

10. Calculations

- 10.1 Measure and record the peak heights of all samples and standards in the order in which they were analyzed.
- 10.2 Use the data for the standards to determine a calibration curve using a linear least squares program.
- 10.3 Calculate the sample concentrations from their peak heights. Multiply by any dilutions made during prep and/or analysis.

11. Reporting

- 11.1 Reporting units are mg/L for water samples and mg/kg for soil samples.
- 11.2 Samples less than the 0.01 mg/L (1 mg/kg) are reported as N.D.
- 11.3 Significant Figures:

Range	Water Samples	Soils/Wastes
0.01 - 0.09	1	----
0.10 - 0.99	2	----
1.0 - 9.9	2	1
10 - 99	2	2
100+	3	3

11.4 LDMS Data Entry

- 11.4.1 The usual standards for data entry apply.

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12. References

12.1 Method source: EPA Methods 420.1, 420.2

12.2 Deviations from source method and rationale.

12.2.1 The two source methods were combined to allow manual distillation and automated colorimetric analysis.

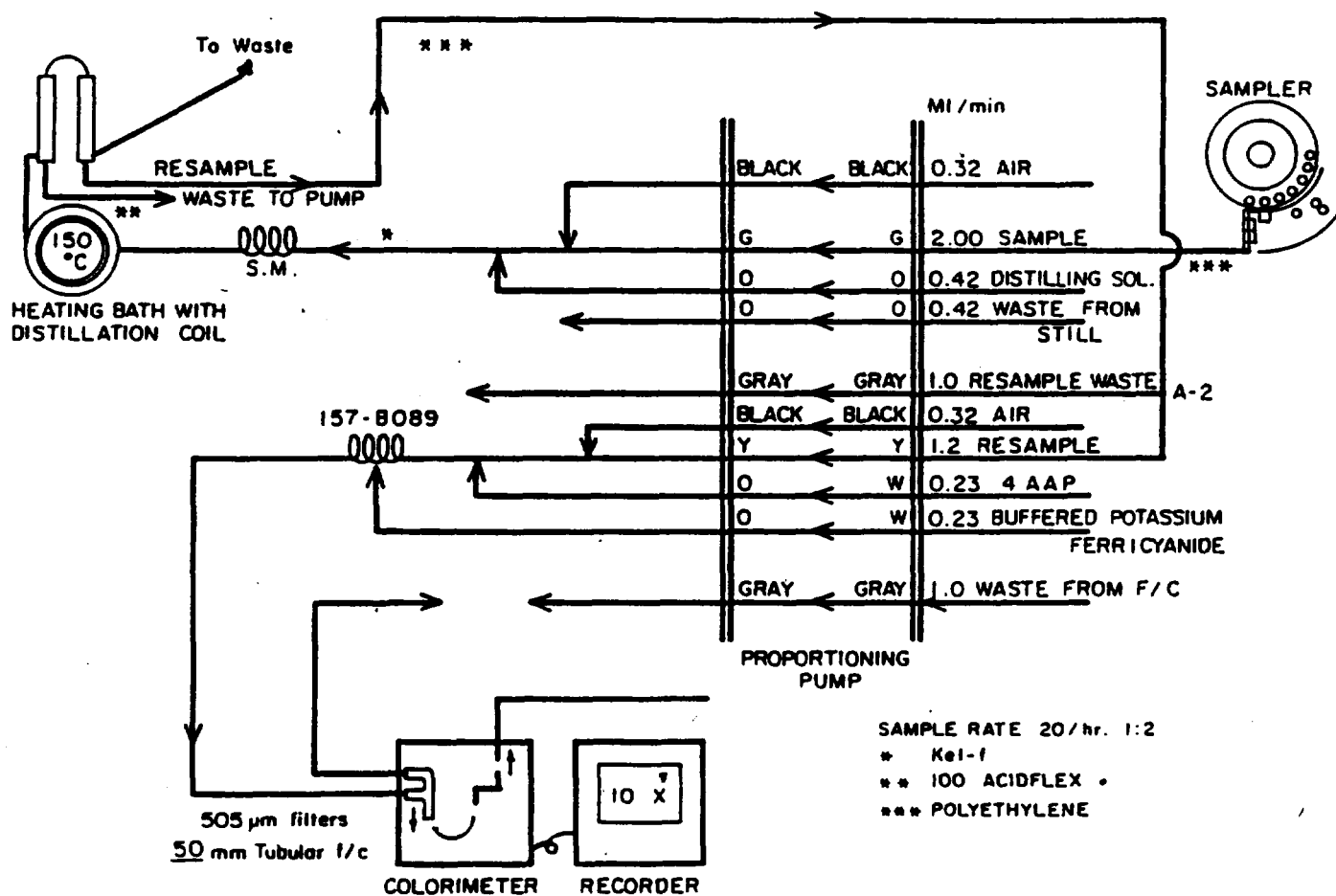
12.2.2 There is a discrepancy between the preservation methods and holding times given in the method and those given in the table of containers and preservatives at the front of the methods book. We have chosen to use sulfuric acid to adjust the sample pH to 2 since this has been done traditionally at RMAL.

12.2.3 The size of the distillation apparatus and volumes of sample and reagent were reduced to conserve space and speed up the analysis.

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Figure 1. Automated Phenol Analysis Schematic



B7

GRAIN SIZE DISTRIBUTION

LABORATORY METHOD STANDARD OPERATING PROCEDURE

Section: Geotechnical	Section Number 9	Revision # 1	Effective Date 12-03-93
SOP Description: Particle Size Analysis (Hydrometer)	SOP # 903	Document No. [soilsop-903]	Page 1 of 7
Reviewed By: C. Scherfer	Approved By: G. Gerry		

PARTICLE SIZE ANALYSIS (HYDROMETER)

Scope and Application

This method is applicable to the determination of particle distribution of soils finer than the No. 200 (0.075mm) U.S. Standard sieve by a sedimentation process.

Method Summary

This test method covers the quantitative determination of the distribution of particle sizes in soils. The distribution of particle sizes larger than 0.075 mm (retained on No. 200 sieve) is determined by mechanical sieving, while the distribution of particle sizes smaller than 0.075 mm is determined by a sedimentation process using a hydrometer.

Method Reference

"Annual Book of ASTM Standards", July 1993, Method D 422-63.

Reporting Limit

0.1%

Sample Handling

For fine-grained soils, collect a minimum of 500g of sample and place in an appropriate container.

Reagents and Apparatus

1. Balance, 0.01g sensitivity
2. Stainless steel spoon or spatula
3. Aluminum pans

LABORATORY METHOD STANDARD OPERATING PROCEDURE

Section: Geotechnical	Section Number 9	Revision # 1	Effective Date 12-03-93
SOP Description: Particle Size Analysis (Hydrometer)	SOP # 903	Document No. [soilsop-903]	Page 2 of 7

4. Air Dry Oven, capable of maintaining 60 ± 5 °C
5. Drying oven, capable of maintaining 110 ± 5 °C
6. Dispersing solution
7. Deionized water
8. Hydrometer, ASTM 152H
9. Sedimentation cylinder, 1000 mL (with 1000 mL mark and rubber stopper)
10. Thermometer, 0.1°C sensitivity
12. Sieve shaker fitted with appropriate sieve sizes
12. Clock/Timer, capable of reading to seconds
13. Mortar and Pestle
14. Glass jar and funnel
15. Squirt bottle, containing deionized water
16. Squirt bottle, containing rubbing alcohol

Reagent Preparation

1. Dispersing solution (hexametaphosphate):
 - a. Tare bowl on scale to 0.00.
 - b. Weigh out 160g of sodium hexametaphosphate.
 - c. Pour into 4 liter bottle using a funnel.
 - d. Weigh 35g of sodium bicarbonate and pour into the 4 liter bottle.
 - e. Fill bottle with deionized water to 4 liter mark.
 - f. Place bottle on top of air-dry oven to dissolve.
 - g. Pour solution into a plastic container when completely dissolved.
 - h. To calibrate for hydrometer correction: Pour 125 mL of the solution into a 1000 mL cylinder. Add deionized water to the 1000 mL mark and seal with rubber stopper.
 - i. Invert cylinder several times to mix.
 - j. Place hydrometer in cylinder. Reading should be 6 ± 1 . This is the hydrometer correction for the worksheet (-6).
 - k. Store dispersing solution at room temperature.

Notes:

1. Room must be maintained at a constant temperature during hydrometer analysis.
2. Refer to Attachment I for example worksheet.

LABORATORY METHOD STANDARD OPERATING PROCEDURE

Section: Geotechnical	Section Number 9	Revision # 1	Effective Date 12-03-93
SOP Description: Particle Size Analysis (Hydrometer)	SOP # 903	Document No. [soilsop-903]	Page 3 of 7

Procedure**DAY 1**

1. Weigh pan (tare) to nearest 0.1g and record pan number.
2. Place representative sample in pan (approximately 500g for fine grained soils).
3. Place soil and pan in air-dry oven (60°C) overnight (12 - 16 hours).

DAY 2

4. If necessary, place representative portion of sample into a mortar and crush sample with pestle. (Note: Do not crush the stones.)
5. Pour sample through No. 10 sieve.
6. Record pan number on jar with a wax pencil. Put glass jar and funnel on scale and tare together. (Make sure scale reads 0.00.)
7. Pour material passing No. 10 sieve into jar until 50g material is obtained for clay and silt soils (75-100g for sandy soils).
8. Record the weight of material in the jar the nearest 0.01g on the worksheet. This is the "P10 Soil Set".
9. Place any remaining material from the mortar and/or No. 10 sieve in original pan. Weigh and record weight of remaining soil and pan to nearest 0.01g (wet weight) on the worksheet.
10. Place pan in 110 ± 5 °C oven overnight (12-16 hours).
11. Pour exactly 125 mL of dispersing solution into jar containing the P10 sample. Shake vigorously and let soak overnight (at least 16 hours). Note: To further help dispersing the soil, shake jar periodically.

DAY 3

12. Take pan out of oven and weigh. Record weight to the nearest 0.01g (dry weight).

LABORATORY METHOD STANDARD OPERATING PROCEDURE

Section: Geotechnical	Section Number 9	Revision # 1	Effective Date 12-03-93
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13. Cover soil with water and let soak in pan. To aid in dispersing soil, add a small amount of dispersing solution to soil and water while soaking.
14. Pour the contents of the jar into a 1000 mL cylinder. Rinse jar and cap with deionized water (into the 1000 mL cylinder), making sure to get all the soil out of the jar and cap. Note: If soil is clumping, pour jar sample into a malt mixer, and add deionized water as needed and mix for one minute. Then pour into the 1000 mL cylinder.
15. Add deionized water until the bottom of the meniscus is at the 1000 mL mark.
16. Make sure form is filled out with times for readings; generally 5 minutes apart per sample.
17. Place rubber stopper on cylinder.
18. Shake cylinder one minute by turning cylinder upside down and back for one minute, approximately one turn per second. Note: Make sure no soil is sticking to the bottom of the cylinder.
19. Set down the cylinder after one minute and wash off stopper with squirt bottle, if necessary.
20. Start time clock immediately after setting down cylinder.
21. Squirt rubbing alcohol in cylinder if foaming occurs. BE CONSERVATIVE when adding the deionized water and alcohol so as not to change the 1000 mL volume by more than 5 mL.
22. Place the hydrometer in the cylinder slowly and gently about 20 seconds before each reading.
23. Record hydrometer reading to the nearest 0.5 grams of soil colloids per liter. It is critical to take readings during the first 15 minute at precise times (\pm 30 seconds). Record times and hydrometer readings on the worksheet.
24. Take readings at: 1, 3, 5, 15, 60, 120, 300, 420, and 1440 minutes.

LABORATORY METHOD STANDARD OPERATING PROCEDURE

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25. Take temperature readings after each hydrometer reading. Note: Since the worksheet is set up with one column for temperature per 3 samples, place the thermometer in one of the cylinders, but take it out while taking hydrometer readings.
26. Hydrometer correction should be -6. Refer to reagent preparation for further detail.

DAY 4

27. After the 24 hour (1440 minutes) reading, wash the hydrometer sample through the No. 200 sieve along with the corresponding pan sample that was soaking.
28. Continue with steps 1-4 of the Sieve Analysis SOP (SOP# 905).

Quality Control

1. Laboratory duplicates will be run at a frequency of 1 per week, unless a Quality Assurance Project Plan (QAPjP) exists for a particular project and dictates otherwise. Duplicate results should be within 10% RPD or corrective action shall be taken.

Calculation

1. Refer to calculation on worksheet attached.
2. The grain size distribution and graph can be calculated using the Grain Size computer program (see program manual for program input and output).
3. Duplicate calculation: $RPD = |Value1 - Value2| / ((Value1 + Value2) / 2) \times 100$

LABORATORY METHOD STANDARD OPERATING PROCEDURE

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Attachment I.

LABORATORY METHOD STANDARD OPERATING PROCEDURE

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[QA_ADMIN-SOILSOP-903]

LABORATORY METHOD STANDARD OPERATING PROCEDURE

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SOP Description: Sieve Analysis Through No. 200 Sieve	SOP # 905	Document No. [soilsop-905]	Page 1 of 5
Reviewed By: C. Scherfer	Approved By: G. Gerry		

SIEVE ANALYSIS THROUGH No. 200 SIEVE

Scope and Application

This method is used to determine the distribution of particle sizes in soils through the No. 200 (0.075 mm) U.S. Standard sieve by mechanical means.

Method Summary

Samples are dried, weighed, soaked in water, washed, dried, and then passed through a series of sieves.

Method Reference

"Annual Book of ASTM Standards", July 1993, Methods D 422-63 and C 136.

Reporting Limit

0.1%

Sample Handling

Collect a minimum of 500g of sample and place in an appropriate container.

Reagents and Apparatus

1. Balance, 0.01g sensitivity
2. Stainless steel spoon or spatula
3. Aluminum pans
4. Drying oven, capable of maintaining 110 ± 5 °C
5. Sieve shaker fitted with appropriate size sieves
6. Tap water

LABORATORY METHOD STANDARD OPERATING PROCEDURE

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7. Wire brush and soft wire brush
8. Dispersing solution

Reagent Preparation

1. Dispersing solution:
 - a. Tare bowl on scale to 0.00.
 - b. Weigh out 160g of sodium hexametaphosphate.
 - c. Pour into 4 liter bottle using a funnel.
 - d. Weigh 35g of sodium bicarbonate and pour into the 4 liter bottle.
 - e. Fill bottle with deionized water to 4 liter mark.
 - f. Place bottle on top of air-dry oven to dissolve.
 - g. Pour solution into a plastic container when completely dissolved.
 - h. To calibrate for hydrometer correction: Pour 125 mL of the solution into a 1000 mL cylinder. Add deionized water to the 1000 mL mark and seal with rubber stopper.
 - i. Invert cylinder several times to mix.
 - j. Place hydrometer in cylinder. Reading should be 6 ± 1 . This is the hydrometer correction for the worksheet (-6).
 - k. Store dispersing solution at room temperature.

Notes

1. For cohesionless soils, refer to ASTM C136 for minimum weights of sandy or aggregate samples.
2. Refer to Attachment I for example worksheet.

Procedure

1. Record pan number and weight of pan (tare) to the nearest 0.01g. Place approximately 500g of representative soil in pan. Dry in oven set at 110 ± 5 °C overnight (12-16 hours). Weigh dried sample and pan (dry weight) and record weight to nearest 0.01g.
2. Cover sample with water. If necessary, add some dispersing solution. Let soak a few hours, preferably overnight.
3. Wash soaked sample over a No. 200 wash sieve until wash water is clear.

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- Put sample back in pan and dry in oven at 110 ± 5 °C until thoroughly dry.
- Pour dry sample through the standard series of sieves consisting of: Nos. 4, 8, 10, 20, 30, 40, 50, 80, 100, 200, and catch pan. Note: The present sieve shaker is fitted for No. 4 sieve on down. If the sample has material larger than No. 4 sieve, hand shake on sieves above No. 4.
- Place both covers on top sieve and shake for approximately 10 minutes, record the weight of the material retained from first sieve and mark "0" for sieve on sieve worksheet above where 100% material passed.
- Continue to weigh material from each sieve cummulatively, making sure the sieves are cleaned with appropriate brushes and pressure. Note: If a sieve has "0" retained and the sieve above it had material retained, simply put a slash for the weight of the sieve.
- Discard material in catch pan (passing No. 200). Save the remaining material until report is complete.

Quality Control

- Laboratory duplicates will be run at a frequency of 1 per week, unless a Quality Assurance Project Plan (QAPjP) exists for a particular project and dictates otherwise. Duplicate results should be within 10% RPD or corrective action shall be taken.

Calculation

- Calculate Percent Finer as follows (Calculate for each sieve size.):

$$\% \text{ Finer} = \frac{(\text{Dry sample weight} - \text{tare}) - (\text{Cumulative weight retained})}{(\text{Dry sample weight} - \text{tare})} \times 100$$

- The grain size distrubution and graph can be calculated using the Grain Size computer program (see program manual for program input and output).
- Duplicate calculation: $RPD = |Value1 - Value2| / ((Value1 + Value2) / 2) \times 100$

LABORATORY METHOD STANDARD OPERATING PROCEDURE

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Attachment I

LABORATORY METHOD STANDARD OPERATING PROCEDURE

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1. 12-03-93

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B8

TOTAL ORGANIC CARBON

QUALITY CONTROL LIMITS FOR THE ANALYSIS OF TOTAL ORGANIC CARBON BY RMT

Provided in this appendix is the standard operating procedure for the analysis of Total Organic Carbon (TOC) on soil and sediment. The detection limit for this analysis is 100 mg/Kg. The following table lists frequency and performance standards for QC samples. If the QC check samples do not meet performance standards, the samples are to be reanalyzed. If a duplicate or spike does not meet the specified limits, the sample will be reanalyzed immediately. If the reanalysis still does not meet the specified limits, the affected data will be flagged. When the concentration exceeds the calibration range, reanalysis of the prepared sample at the appropriate dilution is required.

TOTAL ORGANIC CARBON QC REQUIREMENTS

<u>AUDIT</u>	<u>FREQUENCY*</u>	<u>LIMITS</u>
Lab Blank	1 per 10 samples	< Detection Limit
Check Standard	1 per 10 samples	90 - 110 % Recovery
EPA QC Reference Standard	1 per set	80 - 120 % Recovery
Lab Duplicate	1 per 10 samples	20 RPD (\pm DL if sample concentration is < 5 x DL)
Matrix Spike	1 per 10 samples	75 - 125 % Recovery

*Frequency refers to each matrix individually.

STANDARD OPERATING PROCEDURE

INSTRUMENT TYPE: Total Organic Carbon (TOC) Analyzer
MANUFACTURER: Rosemount Analytical, Dohrmann Division
MODEL: DC-80
LABORATORY NO.:

SERIAL NO.:	DC-80 Electronics Module	Serial No. HD 1264
	DC-80 Reaction Module	Serial No. HD 1291
	ASM-1 Auto Sampler Module	Serial No. HD 1165
	PRG-1 Purgeables Module	Serial No. None
	DC-80 Sludge/Sediment Sampler	Serial No. None
	Printer	Serial No. None

PROCEDURE:

The DC-80 TOC Analyzer can be configured several ways. This procedure will explain the configuration for the analysis of waters (WTOC) and soils (STOC) and will be dealt with in separate sections.

I. Waters TOC (WTOC) configuration

A. Routine Operation

1. Turn on O₂ purge gas tank and adjust line pressure to 30 psi.
2. Turn power switch of ASM-1 autosampler on and verify that "Manual" switch is lit.
3. Turn on Power switch of DC-80 Reaction Module and verify that "Pump" and "Lamp" switches are activated.
4. Turn on Power to DC-80 Electronics Module.
5. Power of Horiba PIR-2000 detector is always left in the "ON" position.
6. Connect junction between manifold ports #4 and #5 located on the right side of the DC-80 Reaction Module. Verify purge gas is bubbling through the reaction vessel.

7. Put pump fingers in place on peristaltic pump on right side of DC-80 Reaction Module. Verify that waste lines feed into proper container and oxidant reservoir is filled.
8. Place function switch on front panel of Electronics Module to "TOC" mode.
9. Place detector function switch on front panel of Electronics Module to "Det" setting. Allow baseline reading to stabilize to historical value, typically 0.0100 - 0.0200.
10. Set 3 function range switch to 1 mL for low level analyses or 200 μ L for high level analyses.
11. Calibrate with appropriate standard based on range selected.

B. Shutdown

1. Remove junction between ports #4 and #5.
2. Turn off purge gas flow.
3. Turn off power switch on front panel of the Electronics Module.
4. Turn off power switch on front panel of the Reaction Module.
5. Turn off power switch on ASM-1 autosampler module.
6. Release tension on peristaltic pump fingers.

II. Soils TOC (STOC) Configuration

A. Routine Operation

1. Turn on O₂ purge gas tank and verify line pressure of 30 psi.
2. Connect lines from PRG-1 purgeables module labeled "4" and "5" to ports "4" and "5," respectively on the right side of the DC-80 Reaction Module. Verify gas flow by observing bubbling in reaction vessel.

3. Turn on power switch labeled "Furnace" on PRG-1 module.
4. Turn "Power" of ASM-1 autosampler module on. Verify that "Manual" switch is lit.
5. Turn "Power" switch of DC-80 reaction module on. Turn off switches labeled "Pump" and "Lamp."
6. Turn on "Power" switch of DC-80 Electronics Module.
7. Set function switch to "TOX."
8. Set injection volume control to "40 μ l" setting.
9. Allow furnace to heat up (~ 30 minutes) and baseline value to stabilize to historical value, typically 0.0100 - 0.0200.
10. Calibrate and operate as described in analytical method for soils TOC. (See RMT Method, Section No. 2.44.)

B. Shutdown

1. Disconnect lines to ports #4 and #5.
2. Turn off purge gas.
3. Turn off power switches on DC-80 Electronics Module, (leave power to Horiba PIR-2000 on at all times), DC-80 Reaction Module, ASM-1 Autosampler Module and PRG-1 Furnace Module.

III. Routine/Preventative Maintenance

- A. Septum Replacement
Replace the injection septum every 100 injections or at any time leakage is obvious.
- B. Reagent Replenishment
Check daily.
- C. Sparge/Carrier Gas Replenishment
Check once a week.

- D. Tin Scrubber
Check daily. Color will change as it is used. Repack the tube when one-half of the tin is exhausted. Use 20 mesh granular tin.
- E. Pump Tube Replacement
Replace every two weeks if instrument is operated continuously. Release pressure fingers when not in use. Plug reagent lines before releasing.
- F. Printer Tape
Check every two or three days. Always check before an unattended automated run.
- G. Infra-red Zero
Check once or twice a day to see that zero reading is around 0.0100 on the digital readout when the detector/ppm switch is in the detector position. It should always be ABOVE zero. A reading of 0.0100 on the DVM is reasonable.
- H. Infra-red Span
The IR span should not need any routine resetting.

REVIEWED BY: Eric L. Thomas
Eric L. Thomas
Supervisor
Inorganic Section

6/1/90
Date

Mark A. Wirtz
Mark S. Wirtz
Quality Control Coordinator

6/1/90
Date

APPROVED BY: R. Alan Doughty *for*
R. Alan Doughty
Laboratory Director

6-4-90
Date



RMT METHOD
SECTION NO. 2.44
REVISION NO. 1
DATE: April 1990
PAGE 1 OF 5

ANALYTICAL METHOD

TITLE: Total Organic Carbon Analysis

DEPARTMENT: Inorganic - Wet Chemistry

APPLICATION: Water and wastewater for nonpurgeable organic carbon

REFERENCE: EPA 600 4-79-020, Revised March 1983, Method 415.2
EPA Manual SW-846, 3rd Edition, Method 9060
Standard Methods for the Examination of Water and Wastewater, 17th Edition, 1989, Method 5310C
Dohrmann DC-80 TOC Systems Manual, Edition 6, January 1984

PROCEDURE SUMMARY:

This procedure uses the persulfate ultraviolet oxidation method. It is a rapid and precise method for the measurement of trace levels of organic carbon in water. Organic carbon is oxidized to CO₂ by persulfate in the presence of ultraviolet light. The CO₂ produced is measured directly by an infrared analyzer.

REVIEWED BY: Eric L. Thomas
Eric L. Thomas
Supervisor
Inorganic Section

4/21/90
Date

Mark S. Wirtz
Mark S. Wirtz
Quality Control Coordinator

4/23/90
Date

APPROVED BY: R. Alan Doughty
R. Alan Doughty
Laboratory Director

4/29/90
Date

SAMPLE HANDLING & PRESERVATION:

The sample is preserved at the time of sampling by acidifying to $\text{pH} \leq 2$ with sulfuric acid (H_2SO_4). The sample is refrigerated in a glass bottle. Hold time for the preserved sample is 28 days.

INTERFERENCES:

The presence of chloride in high concentrations ($> 0.10\%$) interferes with the rate of oxidation. The tailing which results may fall outside of the 8 minute analysis window. The reagent can be slightly modified by adding mercuric chloride and mercuric nitrate. The chloride then complexes with the mercury which in turn allows the carbon to oxidize at a normal rate. The procedure can be found on page 7-1 of the systems manual.

Highly suspended solids can also give variable results. However, it is imperative that the sample be suspended evenly if total organic carbon is being measured. Settling should not occur before injection. Particles may clog the autosampler tubing. Therefore, samples with suspended matter must be manually injected.

APPARATUS:

Dohrmann DC 80 Total Organic Carbon Analyzer
18 x 150 mm test tubes
Syringes: 50 μL , 100 - 250 μL , 1 mL
Volumetric flasks: 100 mL, 200 mL, 1000 mL

REAGENTS:

Deionized (D.I.) water
Potassium persulfate
Nitric acid (HNO_3)
Mercuric chloride (HgCl_2)
Potassium hydrogen phthalate (KHP)
Mercuric nitrate, monohydrate ($\text{Hg}_2\text{NO}_3 \cdot \text{H}_2\text{O}$)

Prepare Potassium Persulfate Solution

- Dissolve 40 grams potassium persulfate in 1 liter of D.I. water.
- Add 1 mL conc. nitric acid. Mix well. Store in cool dark place. Shelf life is one month.

Prepare Potassium Persulfate-Mercuric Salt Solution

- Dissolve 8.2 grams HgCl_2 and 9.6 grams $\text{Hg}_2\text{NO}_3 \cdot \text{H}_2\text{O}$ in 400 mL D.I. water.
- Add 20 grams potassium persulfate and 5 mL conc. nitric acid. Mix well and bring to 1 liter with D.I. water.

Prepare Standard (ppm as Carbon), 2000 ppm

- Dissolve 425 mg KHP in 100 mL D.I. water. Add 0.1 mL conc. HNO_3 . Store in an amber bottle and refrigerate. Shelf life is one month.

Prepare Standard (ppm as Carbon), 400 ppm

- Dilute 20.0 mL 2000 ppm Standard to 100 mL in a volumetric flask. Store in an amber bottle and refrigerate. Shelf life is one month.

Prepare Standard (ppm as Carbon), 10 ppm

- Dilute 1.0 mL 2000 ppm Standard to 200 mL in a volumetric flask. Store in amber bottle, refrigerate. Shelf life is one month.

PROCEDURE:

1. Start-up instrument. (See RMT SOP, Section No. 2.07.)
2. Calibrate the instrument. The instrument is calibrated with a one point standard. When the CAL light is off, the instrument has no calibration in its memory. To calibrate:
 - a. Inject a standard.
 - b. Push START.
 - c. When READY light comes on, push CAL. CAL light should now be on.

When CAL light is on, the instrument is already storing a calibration. In most cases an update of the existing calibration is sufficient. Follow the same calibration steps to update a calibration. The CAL light should remain on the entire time.

In order to erase an existing calibration from memory, simply hold CAL in for at least 1 second. The CAL light will go off.

Select a calibration standard from the following ranges:

<u>Sample Conc.</u>	<u>Volume Injected (uL)</u>	<u>Calibration Std.</u>
0.1 - 20 ppm	1000	10 ppm KHP
10 - 800 ppm	200	400 ppm KHP
100 - 4000 ppm	40	2000 ppm KHP

When selecting a range, make sure the selector knob on the front panel is in the correct position.

3. Sample is prepared for injection. Sample is shaken vigorously and poured into a labeled test tube. (All sediment must be suspended evenly.)
4. Sample is sparged for 10 minutes with oxygen. A syringe is used to agitate the sample to insure uniform suspension of solids. The syringe is then filled to sample volume.
5. Sample is injected and START is pushed. When the sample has been processed, the READY light will come on and the integrated concentration will be sent to the printer. This indicates that the instrument is ready for the next injection.
6. Shut down instrument. (See RMT SOP, Section No. 2.07.)

Quality Control

A Method Blank and calibration standard are run each day to verify calibration. Recalibration (or updating an existing calibration) is necessary if standards vary by more than 10% from calibration. A check standard is run after every 10 samples to monitor system stability.

- Initial Calibration Verification (ICV)
The ICV must be run immediately after calibration and meet current control limits.
- Initial Calibration Blank (ICB)
The ICB must be analyzed after the ICV and be less than the instrument detection limit (IDL).
- Laboratory Control Sample (LCS)
An LCS consisting of known concentration must be prepared and analyzed for each matrix type and meet current control limits.
- Continuing Calibration Verification (CCV)
The CCV is analyzed after every 10 analytical samples and meet current control limits.

Continuing Calibration Blank (CCB)

The CCB is analyzed after every CCV and be less than the IDL.

Spike (10 ppm)

A spike must be performed on each group of samples of a similar matrix type with a frequency of 10%.

Duplicate

A duplicate must be analyzed on each group of samples of a similar matrix type with a frequency of 10%.

Calculations

Spike:

$$\% \text{ Recovery} = \frac{\text{SSR} - \text{SR}}{\text{SA}}$$

SSR - Spiked Sample Result

SR - Sample Result

SA - Spike Added

Duplicate:

If the sample value and/or the duplicate value is less than 5 times the instrument detection limit (IDL), use absolute difference.

$$|\text{Sample} - \text{Duplicate}| = \text{Absolute Difference}$$

If both the sample value and the duplicate value are equal to or greater than 5 times the IDL, use relative percent difference (RPD).

$$\text{RPD} = \frac{S - D}{(S + D)/2} \times 100$$

S - Original Sample Value

D - Duplicate Sample Value



LABORATORIES

Appendix

APPLICATION: To analyze soil, sludge, and solid waste for organic carbon.

REFERENCE: ASTM Method D4129-82, 1982.
Dohrmann DC-80 TOC Systems Manual, ed. 6, January 1984.

SAMPLE HANDLING AND PRESERVATION:

The sample is not preserved, but is refrigerated in a glass bottle.

APPARATUS:

Dohrmann DC 80 Total Organic Carbon Analyzer with Sludge and Sediment
Sample Accessory
Forceps
Watch Glass
50 uL Syringe
Small Spatula
Platinum Boat

PROCEDURE:

1. Start up:

Three POWER 's are turn on--right to left. Furnace is turned on. Gas supply is turned up to 30 psi oxygen. The furnace must be allowed to warm up for approximately one hour. During this time, the two tubes from the furnace should be immersed in water, ph - 10, to absorb any NO_x that may be formed. The furnace is ready when the tube near the sample inlet is glowing. When the baseline is stabilized around 0.0100, the instrument is ready for the first injection. The PUMP and LAMP do not have to be turned on.

The teflon loop is removed form inlets 4 and 5 on the reaction module and lines 4 and 5 from the furnace are attached in its place.

2. Boat preparation:

The platinum boat is lined with quartz wool. The boat is introduced into the furnace and allowed to "bake-out".

3. The instrument is calibrated using 2000 ppm KHP standard. The standard is injected into the boat through a septa in the sample port.

4. Sample preparation:

Sample is mixed until homogeneous.

Transfer approximately 5 grams of sample into a porcelain dish. Add 5% sulfuric acid dropwise, while mixing, until effervescence is no longer visible. Dry in an oven at 105°C until constant dry weight is obtained.

RMT^{INC.}
LABORATORIES

Sample is weight into a lined platinum boat. (Sample size must be kept between 10 and 100 mg.)

Sample is placed in the saddle and the injection port is closed. Sample is allowed to stand outside of furnace for about 2 minutes to stabilize the system.

Note: When calibrating, the boat is immediately introduced into the furnace.

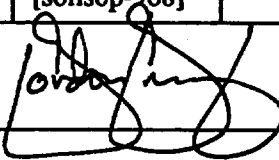
5. **START** is pushed and sample is introduced into the furnace. When the sample has been processed, the **READY** light will come on and the integrated concentration will be sent to the printer. This indicates that the instrument is ready for the next sample.
6. Instrument shut down:

Three **POWER** 's are turned off--left to right. Gas supply is turned off. The reagent supply tube, the two waste exit tubes, and lines 4 and 5 are disconnected to prevent syphoning.

B9

HYDRAULIC CONDUCTIVITY

LABORATORY METHOD STANDARD OPERATING PROCEDURE

Section: Geotechnical	Section Number 9	Revision # 1	Effective Date 01-26-94
SOP Description: Permeability - Rigid Wall / Falling Head	SOP # 908	Document No. [soilsop-908]	Page 1 of 5
Reviewed By: <i>Cynthia A. Scherger</i>	Approved By: 		

PERMEABILITY - RIGID WALL/FALLING HEAD

Scope and Application

This method covers the determination of permeability in soils using the falling head method. The falling head method is generally used for fine grained soils (fine sands to fat clays).

Method Summary

Hydraulic Conductivity (permeability) is tested on about 7 inches of soil inside of a Shelby tube (or remolded tube). The sample is attached to a standpipe with a head of water applied to it without pressure. Daily readings of water head level in the standpipe are taken to determine the rate of flow through the sample.

Method Reference

"U.S. Army Corps of Engineers Manual", EM-1110-2-1906, VII

Reporting Limit

Report permeability in scientific notation in units of cm/sec.

Sample Handling

Collect samples in a Shelby tube.

Reagents and Apparatus

1. Balance, 0.01g sensitivity
2. Carving knife
3. Drying oven, capable of maintaining 110 ± 5 °C
4. Pipe cutter
5. Caliper

LABORATORY METHOD STANDARD OPERATING PROCEDURE

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6. Cloth filters
7. Rubber rings
8. Rigid wall permeability setup
9. Deionized or tap water
10. Extruder

Reagent Preparation

No reagent preparation is required.

Procedure

1. Use the pipe cutter to cut the bottom 7 inches of the Shelby tube sample. Note: If the tube is disturbed on the end, cut off as little of the disturbed end as possible and measure 7 inches from that point to cut the sample.
2. Carve each end of the tube so the soil is even with the tube.
3. Weigh the tube and sample. Use the caliper to measure the length of the soil sample in the tube. Assume 2.9 inches for the inside diameter of a 3-inch Shelby tube. Record all measurements on the worksheet (Attachment I).
4. Set up the tube sample with a cloth filter on each end secured with a rubber ring and place inside the rigid wall permeability setup.
5. Tighten the bolts and place on the floor in the permeability room.
6. Attach the hose to the top of the setup.
7. Place water in the standpipe. (Make sure the water is flowing through the hose by quickly disconnecting the hose and putting it back on.)
8. Fill the standpipe up again to the top (235 cm).
9. Record the start date, time, and start height (235 cm).
10. Take readings approximately every 24 hours. Record the end date, time and end water level. Fill up standpipe again. Note: For sandier soils, readings may be more frequent than every 24 hours.

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11. After 10 - 12 readings are taken, break down the permeability setup.
12. Weigh the tube with sample.
13. Measure the final length of the sample in the tube with the calipers.
14. Push the sample out of the tube with extruder.
15. Set up the sample for Moisture Content. Refer to the Moisture Content SOP (SOP# 902).
16. Note any voids or irregularities on the sample that may have affected the readings.
17. Clean out the inside of the tube and weigh (tare of mold).

Quality Control

1. Laboratory duplicates will be run at a frequency of 1 per 10 investigative samples or sample set (whichever is more frequent). Duplicate results should be within 10% RPD or corrective action shall be taken.

Calculation

1. Wet Soil = (mold+wet soil weight) - mold weight
2. Complete remaining calculations using the computer program. The following constants are required:

Permeability Head Correction = 3 cm

Sample Diameter = 2.9 inches

Standpipe Diameter = 0.27 inches

Final Moisture = Obtain from Moisture Content Test

3. Duplicate calculation: $RPD = |Value1 - Value2| / ((Value1 + Value2) / 2) \times 100$

LABORATORY METHOD STANDARD OPERATING PROCEDURE

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Revision Dates

1. 01-26-94

[QA_ADMIN-SOILSOP-908]

LABORATORY METHOD STANDARD OPERATING PROCEDURE

Section: Geotechnical	Section Number 9	Revision # 1	Effective Date 01-26-94
SOP Description: Permeability - Rigid Wall / Falling Head	SOP # 908	Document No. [soilsop-908]	Page 5 of 5

Attachment I

Falling Head or Pressure Permeability Test

Job No. _____		Date _____		Sheet _____ of _____		Technician _____		Checked By _____	
Project: _____					Sample: _____				

<p>*Initial & Final Density</p> <table border="1" style="width: 100%; border-collapse: collapse;"> <thead> <tr> <th></th> <th>Initial</th> <th>Final</th> </tr> </thead> <tbody> <tr><td>Mold & Wet Soil</td><td>_____</td><td>_____</td></tr> <tr><td>Tare of Mold</td><td>_____</td><td>_____</td></tr> <tr><td>Wet Wt of Soil</td><td>_____</td><td>_____</td></tr> <tr><td>Volume of Mold</td><td>_____</td><td>_____</td></tr> <tr><td>Wet Density, PCF</td><td>_____</td><td>_____</td></tr> <tr><td>% Moisture</td><td>_____</td><td>_____</td></tr> <tr><td>Dry Density, PCF</td><td>_____</td><td>_____</td></tr> <tr><td>% Compaction</td><td>_____</td><td>_____</td></tr> <tr><td>Sample Ht (L)</td><td>_____</td><td>_____</td></tr> <tr><td>Change In (AL)</td><td>_____ 0</td><td>_____</td></tr> <tr><td>Sample Dia (D)</td><td>_____ IN _____ CM</td><td>_____</td></tr> <tr><td>Stand Pipe Dia (d)</td><td>_____ IN _____ CM</td><td>_____</td></tr> </tbody> </table>		Initial	Final	Mold & Wet Soil	_____	_____	Tare of Mold	_____	_____	Wet Wt of Soil	_____	_____	Volume of Mold	_____	_____	Wet Density, PCF	_____	_____	% Moisture	_____	_____	Dry Density, PCF	_____	_____	% Compaction	_____	_____	Sample Ht (L)	_____	_____	Change In (AL)	_____ 0	_____	Sample Dia (D)	_____ IN _____ CM	_____	Stand Pipe Dia (d)	_____ IN _____ CM	_____	<p>*Initial & Final Moisture Contents</p> <table border="1" style="width: 100%; border-collapse: collapse;"> <thead> <tr> <th></th> <th>Initial</th> <th>Final</th> </tr> </thead> <tbody> <tr><td>Pan No.</td><td>_____</td><td>_____</td></tr> <tr><td>Pan & Wet Soil</td><td>_____</td><td>_____</td></tr> <tr><td>Pan & Dry Soil</td><td>_____</td><td>_____</td></tr> <tr><td>Tare of Pan</td><td>_____</td><td>_____</td></tr> <tr><td>% Moisture</td><td>_____</td><td>_____</td></tr> <tr><td>3" S.T.</td><td>_____</td><td>_____</td></tr> <tr><td>Proctor Mold Number</td><td>_____</td><td>_____</td></tr> <tr><td>Other</td><td>_____</td><td>_____</td></tr> <tr><td>Maximum Density</td><td>_____ lb/cu. ft.</td><td>_____</td></tr> <tr><td>Optimum Moisture</td><td>_____ %</td><td>_____</td></tr> <tr><td>Sample Length (L)</td><td>_____ IN</td><td>_____ CM</td></tr> <tr><td>Sample Area (A)</td><td>_____ IN²</td><td>_____ CM²</td></tr> <tr><td>Permeameter Head Correction</td><td>_____ CM</td><td>_____</td></tr> <tr><td colspan="3">1 PSI = 70.317 CM Water Head</td></tr> </tbody> </table>		Initial	Final	Pan No.	_____	_____	Pan & Wet Soil	_____	_____	Pan & Dry Soil	_____	_____	Tare of Pan	_____	_____	% Moisture	_____	_____	3" S.T.	_____	_____	Proctor Mold Number	_____	_____	Other	_____	_____	Maximum Density	_____ lb/cu. ft.	_____	Optimum Moisture	_____ %	_____	Sample Length (L)	_____ IN	_____ CM	Sample Area (A)	_____ IN ²	_____ CM ²	Permeameter Head Correction	_____ CM	_____	1 PSI = 70.317 CM Water Head		
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☐ Undisturbed or ☐ Remolded Sample

	Date and Time Start T ₁	Date and Time End T ₂	Elapsed Time, Sec. (T)	Pressure PSI (P)	Start Head (H ₁)	End Head (H ₂)	* $K_v = \frac{(d^2)(L)}{(D)^2(T)} \ln \frac{(P)}{(P)-H_2}$
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							
13							
14							
15							
16							

*See computer print out

C


FIELD MEASUREMENT SOPs

- C1 pH
- C2 Conductivity
- C3 Field GC VOC Screen
- C4 PID Headspace Screening

Cl

pH

FIELD SAMPLING AND TESTING SOPs AND TGDs

Section: Groundwater/Leachate Sampling and Testing	Section Number 312	Date of Issue April 1993	Reviewed By S. Wiskes
Subject: Field Measurements pH	Page of 1 5	Date Revised \	Authorized By 

Scope and Application: This method is applicable to surface water, wastewater and groundwater.

Method: Potentiometric

Reference: EPA 1983, p. 150.1

Sensitivity: 0.01 pH unit

Optimum Range: pH 1.00 to 12.00

Sample Handling: Determine on-site, if possible, otherwise within 24 hours.


Reagents and Apparatus:

1. pH meter (Beckman with automatic temperature compensation (ATC))
2. pH electrode and temperature probe
3. Magnetic stirrer and stir bars (for lab use)
4. Beakers or plastic cups
5. pH buffer solutions, pH 4.00, 7.00, and 10.0
6. Deionized water
7. Equipment calibration/return form; or field testing/sample preparation form (Figure 1)

Notes:

1. The pH test is temperature dependent. Beckman pH meters are equipped with automatic temperature control(ATC) and compensate for the differences of temperature between samples and buffers.
2. Interferences in pH measurements occur with presence of weak organic and inorganic salts, and oil and grease. If oil and grease are visible, note on data sheet. Clean electrode with soap and water, followed by 10% HCl and deionized water. Then recalibrate meter before analysis of next sample.

FIELD SAMPLING AND TESTING SOPs AND TGDs


Section: Groundwater/Leachate Sampling and Testing	Section Number 312	Date of Issue April 1993	Reviewed By S. Wiskes
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3. Electrode should be stored in pH 4.00 buffer, or deionized water. While some probes can be stored dry, they work best when stored wet.
4. Before leaving laboratory for field work:
 - a. Check batteries.
 - b. Do a calibration at pH 4.00 and 7.00 to check electrode response and batteries.
 - c. Obtain fresh pH buffer solutions (if necessary).
5. Following field measurements:
 - a. Report any problems with meter or electrode.
 - b. Clean meter and meter case.
 - c. Make sure electrode is stored in pH 4.00 buffer.

Calibration:

1. Turn on the pH meter and clear the previous calibration.
2. Rinse electrode and temperature probe with deionized water and place in pH 4.00 buffer solution, press pH, press standard.
3. Wait for reading to stabilize.
4. Rinse the electrode with DI water then place the electrode and temperature probe in fresh pH 7.00 buffer solution, press standard.
5. Allow meter to stabilize.

FIELD SAMPLING AND TESTING SOPs AND TGDs

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6. Rinse electrode with deionized water and place in pH 7.00 buffer. If meter reading is not 7.00 ± 0.05 , follow Steps 1 through 6 again.
7. If the sample pH is greater than 8, then the meter should be calibrated with pH buffers 7 and 10. The same procedure as described above is followed, this time entering pH 7 first then pH 10.


Procedure:

1. All glassware is to be soap and water washed, tap rinsed and deionized water rinsed prior to analyses (lab).
2. Calibrate meter using calibration procedure. Record the meter used and pH buffers on data sheet (Figure 1).
3. Pour the sample into clean beaker or plastic cup.
4. Place stir bar in beaker and put on magnetic stirrer (low speed) for lab measurement of pH. Swirl cup gently for field measurement of pH.
5. Rinse electrode and temperature probe with deionized water.
6. Immerse electrode and temperature probe in sample. The white KCl junction on side or bottom of electrode must be fully immersed in solution. Allow sufficient time for reading to stabilize. Record sample I.D., pH and time analyzed on a data sheet (Figure 1). Rinse electrode and temperature probe with deionized water.

Quality Control:

1. Duplicate 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate is still required. Duplicates should be within acceptable ranges. Average the results.

FIELD SAMPLING AND TESTING SOPs AND TGDs

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2. Check calibration with pH 7.00 buffer solution initially and after every 10 samples and at the end of the analytical run and record on data sheet (Figure 1). Buffer solution should read ± 0.05 of true value.

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Figure 1

Page 1

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Date

4.8.92

Field Testing/Sampling Preparation

Lab No.	Sample No.	Time Filtered/ Analyzed	pH	Spec. Cond.	Temp.	Spec. Cond. @ 25 deg.	Analyst	Comments
	MW1	11:50	7.52				Stw	

pH Meter: Beckman #10 ^{8:45 AM} pH 4.0 = 4.00
 Conductivity Meter: 70 = 7.03
 Bottle Types/Preservation:
 Conductivity of Standard:


Filter:

15:05

C2

CONDUCTIVITY

FIELD SAMPLING AND TESTING SOPs AND TGDs

Section: Groundwater/Leachate Sampling and Testing	Section Number 313	Date of Issue April 1993	Reviewed By S. Wiskes
Subject: Conductivity YSI Meter	Page of 1 6	Date Revised 1	Authorized By 

Scope and Application: This method is applicable to surface water, wastewater and groundwater.

Method: Specific Conductance (Electrical Conductivity), umhos/cm @ 25°C

Reference: EPA 1983, Method 120.1

Detection Limit: 10 umhos/cm @ 25°C

Sample Handling: Determine on-site

Reagents and Apparatus:

1. Conductivity meter, YSI 33 SCT
2. Deionized water
3. Conductivity standards 1000 umhos/cm and 10,000 umhos/cm @ 25°C. Commercially Available


Notes:

1. All conductivity readings must be corrected to 25°C. This is an office calculation not a field procedure. Therefore, be sure to record the temperature of the sample so that this calculation can be made. See Table 2 for the correction factors.

Procedure:

1. All glassware is to be soap and water washed, tap rinsed and deionized rinsed prior to analysis (lab).
2. With mode switch of the meter in the OFF position. Check the zero setting. If not at zero, use meter adjusting screw to zero (on front of the meter).
3. Turn mode switch to red line, and turn red line knob until needle aligns with red line on dial. Change batteries if meter cannot be aligned.


FIELD SAMPLING AND TESTING SOPs AND TGDs

Section: Groundwater/Leachate Sampling and Testing	Section Number 313	Date of Issue April 1993	Reviewed By S. Wiskes
Subject: Conductivity YSI Meter	Page of 2 6	Date Revised \	Authorized By 

4. Plug probe into jack located on side of meter.
5. Analyze the conductivity standards. If the result is within 90-110% of the standard value, analyze samples. A standard should be analyzed after every 10 samples and at the end of the analytical run. Table 1 lists non-temperature compensated values for both of the standards. Use this table as a reference when a calculator is not available to perform temperature correction on the standards.
6. Totally immerse and suspend the probe in the water sample. Do not allow probe to touch the sides of the sample container.
7. Turn mode switch to the appropriate conductivity scale (X100, X10, or X1). Use the scale that produces a mid-range output on the meter.
8. Wait for needle to stabilize (about 15 seconds) and record conductivity. Multiply the conductivity reading by the scale setting.
9. While gently agitating the probe, take sample temperature (°C) to nearest 0.5°C and record.
10. Rinse probe with deionized water.
11. Record sample I.D., time analyzed, specific conductivity, temperature (to nearest 0.5°), and specific conductivity corrected to 25°C on the data sheet and/or a bound field notebook. See calculation below.
12. Store probe in D.I. water between uses whenever possible.
13. Turn mode switch to the OFF position when finished.

WARZYN INC.

FIELD SAMPLING AND TESTING SOPs AND TGDs

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Subject: Conductivity YSI Meter	Page of 3 6	Date Revised 1	Authorized By 

Quality Control:

1. Quality control calibration standards of 1,000 umhos/cm and 10,000 umhos/cm are to be analyzed, initially and after every 10 samples. If less than 10 samples are analyzed, calibration standards are still required. After the last sample has been analyzed, the calibration standards need to be run to verify calibration. These standards must be within 90-110% of the standard value or the samples run after the last acceptable check standard are to be reanalyzed on a different meter or in the laboratory.
2. Duplicate a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate is still required. Duplicates are to be averaged. Duplicate values are to be within acceptable ranges.

Calculation:

1. Calculate specific conductivity at 25°C using following formula:

$$\text{Cond. @ 25} = \frac{\text{Cond.}}{[1 + 0.02 (T-25)]}$$

Cond. @ 25 = Specific conductivity at 25°C, umhos/cm

T = Temperature of sample, °C

Cond. = Conductivity of sample at temperature T, umhos/cm

FIELD SAMPLING AND TESTING SOPs AND TGDs

Section: Groundwater/Leachate Sampling and Testing	Section Number 313	Date of Issue April 1993	Reviewed By S. Wiskes
Subject: Conductivity YSI Meter	Page of 4 6	Date Revised 1	Authorized By <i>[Signature]</i>

TABLE 1

Non-Temperature Compensated Conductivity
Standard Readings for 1,000 and 10,000 umhos/cm Standards

Conductivity Standard 1,000 umhos/cm			Conductivity Standard 10,000 umhos/cm		
Conductivity Standard Should Read	at	Degrees C°	Conductivity Standard Should Read	at	Degrees C°
500		0	5000		0
520		1	5200		1
540		2	5400		2
560		3	5600		3
580		4	5800		4
600		5	6000		5
620		6	6200		6
640		7	6400		7
660		8	6600		8
680		9	6800		9
700		10	7000		10
720		11	7200		11
740		12	7400		12
760		13	7600		13
780		14	7800		14
800		15	8000		15
820		16	8200		16
840		17	8400		17
860		18	8600		18
880		19	8800		19
900		20	9000		20
920		21	9200		21
940		22	9400		22
960		23	9600		23
980		24	9800		24
1000		25	10,000		25
1020		26	10,200		26
1040		27	10,400		27
1060		28	10,600		28
1080		29	10,800		29
1100		30	11,000		30
1120		31	11,200		31
1140		32	11,400		32
1160		33	11,600		33
1180		34	11,800		34
1200		35	12,000		35

FIELD SAMPLING AND TESTING SOPs AND TGDs

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TABLE 2

Specific Conductivity Temperature Correction

<u>Sample Temperature (C°)</u>	<u>Dermal Correction</u>
1	0.52
2	0.54
3	0.56
4	0.58
5	0.60
6	0.62
7	0.64
8	0.66
9	0.68
10	0.70
11	0.72
12	0.74
13	0.76
14	0.78
15	0.80
16	0.82
17	0.84
18	0.86
19	0.88
20	0.90
21	0.92
22	0.94
23	0.96
24	0.98
25	1.00

Conductivity @ 25° = $\frac{\text{Conductivity}}{\text{decimal correction for sample temp.}}$

Figure 1

Page 1

C# 1000000

Date 4.8.93

Field Testing/Sampling Preparation

Lab No.	Sample No.	Time Filtered/ Analyzed	pH	Spec. Cond.	Temp.	Spec. Cond. @ 25 deg.	Analyst	Comments
.	MW1	1130		500	12°	675	Stw	

pH Meter:

Conductivity Meter: YSI # 1000 std = 760 at 13°, 10,000 std = 7600 at 13°

Bottle Types/Preservation:

Conductivity of Standard:

Filter:

Photo

C3

FIELD GC VOC SCREEN

Effective Date: 1-13-92

FIELD ANALYSIS OF VOLATILE ORGANICS

Scope and Application: This method covers the determination of the following 17 organic compounds in water and soil gas. 4

Target Compounds:

Benzene	trans-1,2-Dichloroethene	Trichloroethene
1,1-Dichloroethane	Ethyl benzene	m-Xylene
1,2-Dichloroethane	Tetrachloroethane	o-Xylene
1,1-Dichloroethene	Toluene	p-Xylene
cis-1,2-Dichloroethene	1,1,1-Trichloroethane	

Note: m-xylene and p-xylene are not separated by this method. Therefore the sum of the unresolved peaks are reported.

Method: Headspace - Gas Chromatographic/Photoionization and Hall Electrolytic Conductivity Detection.

Reference: "EPA Test Methods for Evaluating Solid Waste", SW-846 Methods 3810, 8010 and 8020 with modifications.

Reporting Limits: See table 1

Optimum Range: Headspace 5.0-50 ug/L of water, soil gas 5.0-50 ug/L of soil gas.

Sample Handling: Water samples are to be collected in 40 mL vials with open screw-caps and teflon faced silicone septa. They should be collected so that no headspace remains in the bottle. Soil gas samples are to be collected in 250 mL glass bulbs in a manner that provides the complete purging of the bulb. All samples should be protected from sunlight and transported to the field lab as soon as possible.

Reagents and Apparatus:

1. Open screw cap 40 mL vial (Pierce #13075 or equivalent).
2. Septum - Teflon-faced silicone (Pierce #12722 or equivalent).
3. ~~250 mL gas sampling bulbs.~~
1 L Tedlar bags

4. Gas chromatograph - Varian 3400 equipped with PID and Hall detectors in series.
5. Column 1 - 8-ft x 1/8-in. stainless steel, packed with 1% SP 1000 on Carbopack B (60/80 mesh).
6. Dual-channel Integrator/Recorder.
7. Syringes - 1 and 5 mL gas tight, fitted with shut-off valves and 22 gauge needle. 10, 100, and 1,000 mL gas tight syringes.
8. Balance - ± 0.0001 g.
9. Balance - ± 0.01 g.
10. Reagent water - organic free water or cold tap water which has been shown to be organic free at the method detection limit.
11. 25 mL TC graduated cylinders.
12. Constant temperature water bath - 55°C.
13. Volumetric flasks - assorted.
14. Pipettes - assorted.
15. Standard reference materials → Chem Service
16. Screw top vials - 10 mL
17. Mininert valves caps lined with teflon.

Standard Preparation:

1. **Stock standard solutions:** Prepare a VOC standard containing the target analytes at 5000 ug/mL in methanol.
 - Add about 20 mL of methanol to a 25 mL volumetric flask. Allow the flask to stand unstoppered until the methanol on the neck of the flask has dried. Replace the stopper.
 - Tare the flask on the analytical balance.

- Remove the stopper and, using a 100 uL syringe, add 0.125 g (correct for % purity) of the reference material to the flask. Make sure the drops fall directly into the methanol without contacting the neck of the flask. Replace the stopper.
- Determine the amount of reference material added. Rinse the syringe with methanol, tare the flask, and add the next standard.
- After all the reference materials are added, fill to volume with methanol, cap and invert to mix.
- Transfer the final stock standard into a screwtop vial and cap the using a mininert valve teflon cap.

2. Secondary Standard Solutions

Prepare secondary target standards according to the following scheme:

Standard	mLs	Final Volume	Concentration
5000 ug/mL	1 mL	10 mL	500 ug/mL
500 ug/mL	1 mL	10 mL	50 ug/mL
50 ug/mL	2 mL	10 mL	10 ug/mL

Dilute to the volume with methanol and transfer into a screwtop vial with mini inert valve teflon cap.

Note: Stock standards and secondary standards should be prepared before going out into the field. This will reduce in the necessary equipment needed on-site.

3. Working Headspace Calibration Standards: Prepare working calibration standards according to the following scheme:

Secondary Standard	Amount	Final Volume	Concentration
500 ug/mL	20 uL	200 mL	50 ug/L
50 ug/mL	40 uL	200 mL	10 ug/L
50 ug/mL	20 uL	200 mL	5 ug/L

Fill a 200 mL volumetric flask with reagent water to the mark. Directly inject the secondary standard into the water with an appropriate microliter syringe.

Invert each standard 3 times, discard the first 10 mL in the neck of the volumetric and transfer aliquots of the freshly prepared working standards to 40 mL VOC vials, (No headspace) and cap.

Calibration: Target Headspace Standards

1. Remove and discard approximately 10 mL from a freshly prepared standard and place the vial (capped) now having about 10 mL of headspace in a 55°C water bath with the water level sufficient to equal the water level in the vial.
2. Allow time for equilibration of temperature (10 minutes).
3. Through the septum of the vial, using a 5 mL gas tight syringe with needle remove 5 mL of headspace gas for injection into the gas chromatograph.
4. Construct an external standard curve of peak area response versus concentration for each of the compounds of interest.
5. A calibration check is performed after each set of 10 samples and as the last sample of the day. If the response for any of the target compounds varies from the calculated response by more than $\pm 30\%$, a new calibration curve should be prepared.

Soil Gas (Total Nanograms):

1. Inject 5.0 uL of the 5 ug/mL standard into the gas chromatograph.
2. Use a 1-point standard curve of peak area response versus total nanograms injected for each of the compounds of interest.
3. A calibration check is performed after each set of 10 samples and as the last sample of the day. If the response for any of the compounds varies from the expected response by more than $\pm 30\%$, the average response should be used.

Sample Analysis:

Water Samples:

1. Water samples are received in 40 mL VOC vials. Uncap and decant 10 mL of the sample from the vial. Recap the vial containing 30 mL of sample.
2. Place vials in a 55°C water bath and allow to equilibrate for 10 minutes.
3. Through the septum of the vial, using a 5 mL gas tight syringe with needle remove 5 mL of headspace gas for injection into the gas chromatograph.

4. If any compound of interest is outside the calibration curve and an accurate concentration is required, a dilution of the sample is made using organic free water and a fresh vial of sample. The headspace analysis is then repeated.

Soil Gas Samples:

1. Soil gas samples will be received in ^{1 L Tedlar Bags} ~~250 mL glass bulbs~~. When received, they are allowed to equilibrate to the ambient air temperature. ^{2011 11-20-44}
2. Remove 5 mL of soil gas through the sampling septum and inject into the gas chromatograph.
3. If any compound of interest is outside the calibration curve and an accurate concentration is required, a smaller aliquot is taken from the same sample ~~bulb~~ ^{bag}.

Chromatographic Conditions:

Column: 8-ft x 1/8-in stainless steel, packed with 1% SP-1000 on Carbowax B (60/80 mesh).

Carrier Gas: Helium - Ultra High Purity Grade (Linde) 35 mL/min

Detectors: (in series)

1. Photoionization 10.2eV

Sensitivity - Range 11 x Attenuation 8
Temperature - 240°C

2. Hall 700A

Mode - Halogen
Reactor Temperature - 1000°C
Solvent Flow - 0.8 mL/min Methanol
Hydrogen Flow - 60 mL/min

Injector: Temperature - 200°C

Oven*: Initial - 60° - 0 min
Rate 20°C/min
Final - 200°C - 7 min

* Conditions listed can be varied as needed for changing applications.

Headspace Calculations:

1. Review the chromatograms and data reports for each analysis. Check for gross errors such as incomplete data reports because of faulty integration.
2. Prepare external standard calibration curves for each compound using at least three data points and linear regression analysis.
3. Calculate the concentration found in the samples from the calibration curves using the following equations:

$$\text{ug/L} = A \times \text{DF}$$

where: A = Amount of compound found in the analysis in ug/L (from linear regression). DF = Dilution factor.

Soil Gas ~~Bulb~~ Calculations:

1. Review the chromatograms and data reports for each analysis. Check for gross errors such as incomplete data reports because of faulty integration.
2. Calculate the mass per liter of each parameter found in the samples using the following equation.

$$\text{ng/L} = \frac{R(\text{samp}) \times \text{ng}(\text{std})}{R(\text{std}) \times \text{VL}}$$

where: R(samp) = Response of parameter in sample
R(std) = Response of parameter in standard
ng(std) = ng of standard injected
VL = Volume of aliquot taken from bulb (in L)

Data Reporting:

1. All results, standards conditions, and notes will be recorded in a bound field notebook.
2. All data generated by field G.C. will be considered as tentatively identified, with concentration being estimated.
3. All raw field data will be forwarded to Warzyn Inc., Analytical Laboratory for final review and archiving.

Quality Control:

1. Each analytical run should begin with a headspace standard curve consisting of 50, 10, 5 ppb and a blank. Every eleventh analysis thereafter and the last sample analyzed should be a 10 ppb standard. Continuing calibration standards should be within $\pm 30\%$ of the original standards or a new standard curve should be prepared and samples analyzed since the last check standard reanalyzed.
2. Direct inject 5 μL of a 5 $\mu\text{g/mL}$ target standard (25 ng) for a 1-point soil gas curve.
3. A minimum of 10% duplicate samples should be analyzed. If less than 10 samples are analyzed, a duplicate sample should still be analyzed. Duplicates should be within $\pm 15\%$.
4. New stock standards should be prepared monthly in the laboratory. New secondary standards should be prepared weekly in the laboratory and brought to the field location while maintaining a temperature of approximately 4°C (iced).

TABLE 1**Target VOC Reporting Limits for Water Headspace**

Compound	Reporting Limits (ug/L of water)
Toluene	5.0
1,1-Dichloroethene	5.0
Trans-1,2-Dichloroethene	5.0
Trichloroethene	5.0
Tetrachloroethene	5.0
Benzene	5.0
Ethyl Benzene	5.0
1,1,1-Trichloroethene	5.0
1,1-Dichloroethane	5.0
1,2-Dichloroethane	5.0
cis-1,2-Dichloroethene	5.0
m+p-Xylene	5.0
o-Xylene	5.0

Soil Gas Reporting Limits

Compound	Reporting Limits (ug/L of soil gas)
Toluene	5.0
1,1-Dichloroethene	5.0
cis-1,2-Dichloroethene	5.0
Trans-1,2-Dichloroethene	5.0
Trichloroethene	5.0
Tetrachloroethene	5.0
Benzene	5.0
Ethyl Benzene	5.0
1,1,1-Trichloroethene	5.0
1,1-Dichloroethane	5.0
1,2-Dichloroethane	5.0
m+p-Xylene	5.0
o-Xylene	5.0

SOIL SAMPLE ADDENDUM

FIELD ANALYSIS OF VOLATILE ORGANICS

Scope and Application

This addendum to the SOP "Field Analysis of Volatile Organics" (BC-FGC), as presented in Appendix C3 of the approved June 1992 Beloit QAPP, covers the determination of 17 organic compounds in soils.

Reference

"EPA Test Methods for Evaluating Solid Waste", SW-846 Methods 3810, 8010 and 8020 with modifications.

Reporting Limits

See Table 1.

Optimum Range

Soil 15 to 150 ug/kg of soil.

Sample Handling

Soil samples are to be collected in 4 ounce wide mouth glass jars with teflon seals. The soil should be packed into the jar so that no headspace remains. All samples should be protected from light and transported to the field lab as soon as possible. Samples should be kept cool until they can be analyzed.

Sample Analysis:

Soil Samples:

1. Soil samples are received in 4 ounce wide mouth glass jars. Uncap, and weigh out 10.0 grams of soil into a 40 mL VOC vial. Remove the soil directly against the top of the jar prior to taking sample aliquot.
2. Add 20 mL of reagent grade water and cap the vial with the teflon faced silicon septum.
3. Shake the vial for 15 seconds.

4. Place the vial in a 55°C water bath and allow the sample to equilibrate for 10 minutes.
5. Through the septum of the vial, using a 5 mL gas tight syringe with needle, remove 5 mL of headspace gas for injection into the gas chromatograph.
6. Construct an external standard curve of peak area response versus concentration for each of the compounds of interest.
7. If the sample response for any target compound exceeds the response for the high standard, a smaller aliquot can be taken from a freshly prepared sample.
8. A calibration check is performed after each set of 10 samples and as the last sample of the day. If the response for any of the compounds varies from the expected response by more than $\pm 30\%$, a new calibration curve should be prepared.

Soil Sample Calculations

1. Review the chromatograms and data reports for each analysis. Check for gross errors such as incomplete data reports because of faulty integration.
2. Prepare external calibration curves for each compound using at least three data points and linear regression analysis.
3. Calculate the concentration found in the samples from the calibration curves using the following equation:

$$\text{ug/Kg} = A \times \text{DF}$$

where:

A = Amount of compound found in the analysis in ug/Kg (from linear regression).

DF= Dilution factor. DF is calculated as the ratio of water to soil:

$$\text{DF} = (\text{grams Sample} + \text{mL water}) / \text{grams sample}$$

(e.g., For soil samples prepared using 10.0 grams of soil and 20 mL water, $\text{DF} = ((10.0 + 20) / 10) = 3.$)

JAH/ml/PML

[mad-607-179e]
1526892/15197

TABLE 1**Target VOC Reporting Limits for Soil Headspace**

<u>Compound</u>	<u>Reporting Limits (ug/Kg)</u>
Toluene	15
1,1-Dichloroethene	15
trans-1,2-Dichloroethene	15
Trichloroethene	15
Tetrachloroethene	15
Benzene	15
Ethyl benzene	15
1,1,1-Trichloroethane	15
1,1-Dichloroethane	15
1,2-Dichloroethane	15
cis-1,2-Dichloroethene	15
m+p Xylene	15
o Xylene	15

JAH/vlr/PML
[mad-607-179e]
1526892/15197

C4

PID HEADSPACE SCREENING

FIELD HEADSPACE METHOD FOR PID SCREENING OF SOILS

Following the removal of the split spoon sampler from the borehole, the sampler is opened and a brief observation is made of the lithology of the sample. A representative subsample is removed from the split spoon and is placed in a glass soil jar for field headspace screening.

The field headspace screening jar is filled approximately half way. A piece of aluminum foil is placed over the top of the jar and the jar lid is screwed on. The sample is agitated to break up soil clods and release vapors.

Following equilibration, the photoionization detector (PID) probe is used to pierce the aluminum foil and the PID response is recorded.

In the event that the split spoon sample recovery volume is inadequate to provide the subsample, the sample for inorganic laboratory analysis will be used to perform the field headspace screening.

RJR/vlr/JAH
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Table C4-1

Summary of Chemical And Physical Properties of Organic Non-Aqueous Phase Liquids Detected in Phase I

Beloit Corporation Blackhawk Facility
Rockton, Illinois

COMPOUND	Density (g/cc)	Water Solubility (mg/L)	1% of Solubility (ug/L)	Ionization Potential (eV)	Vapor Pressure (mm Hg)	Henry's Law Constant (atm-m ³ /mole)	Koc (ml/g)	Soil NAPL Criteria Above WT (mg/kg)	Soil NAPL Criteria Fine-grained (mg/kg)	MW (g/mole)	Log Kow (ml/g)
Chloromethane	0.92	6,500	65,000		4,310	4.40E-02	35	573	1,092	50	0.95
Vinyl chloride	1.37	2,670	26,700	10.00	2,660	8.19E-02	57	294	507	63	1.38
Methylene chloride	1.33	20,000	200,000	11.35	360	2.03E-03	8.8	1,240	2,836	85	1.30
Acetone	0.79	1,000,000	10,000,000	9.69	270	2.06E-05	2.2	55,391	135,179	58	-0.24
Carbon disulfide	1.26	2,940	29,400	10.13	360	1.23E-02	54	315	550	76	2.00
1,1-Dichloroethene	1.22	2,250	22,500		600	3.40E-02	65	266	445	97	1.84
1,1-Dichloroethane	1.18	5,500	55,000	11.06	182	4.31E-03	30	458	896	99	1.79
1,2-Dichloroethene	1.26	6,300	63,000	9.70	324	6.56E-03	39	581	1,083	97	0.48
1,2-Dichloroethane	1.25	8,520	85,200	11.04	64	9.78E-04	14	572	1,252	99	1.48
2-Butanone	0.81	268,000	2,680,000	9.53	77.5	2.74E-05	4.5	15,461	36,844	72	0.26
1,1,1-Trichloroethane	1.34	1,500	15,000	11.25	123	1.44E-02	152	308	427	133	2.50
Trichloroethene	1.46	1,100	11,000	9.45	57.9	9.10E-03	126	197	285	131	2.38
Benzene	0.88	1,750	17,500	9.25	95.2	5.59E-03	83	238	378	78	2.12
4-Methyl-2-pentanone	0.8	17,000	170,000	9.30	6.0	1.59E-02	20.5	1,253	2,609	100	0.88
Tetrachloroethene	1.62	150	1,500	9.32	17.8	2.59E-02	364	63	75	166	2.60
Toluene	0.87	535	5,350	8.82	28.1	6.37E-03	300	189	232	92	2.73
Ethylbenzene	0.87	152	1,520	8.76	7.0	6.43E-03	1100	175	187	106	3.15
Total Xylenes	0.9	466	4,660	8.56	10.0	7.04E-03	330	179	216	106	3.26
1,4-Dichlorobenzene	1.46	79	790	8.94	1.18	2.89E-03	1700	139	145	147	3.60
Phenol	1.07	93,000	930,000	8.69	0.341	4.54E-07	14.2	6,267	13,688	94	1.46
1,4-Dichlorobenzene	1.46	79	790	8.94	1.18	2.89E-03	1700	139	145	147	3.60
2-Methylphenol	1.03	30,000	300,000		0.240		500	16,596	18,989	108	1.97
4-Methylphenol	1.02	30,000	300,000		0.110	4.95E-04	500	16,596	18,989	108	1.97
2,4-Dimethylphenol	1.03	4,600	46,000		5.90E-02		42	438	805	122	2.36
Naphthalene	0.96	32	320	8.10	2.60E-04	1.11E-03	649	22	25	128	3.45
2-Methylnaphthalene	1.01	27	270		5.90E-02		712	21	23	142	3.43
Acenaphthene	1.02	3,420	34		1.55E-03	9.20E-05	4600	16	16	154	4.00
Dibenzofuran	1.09	21	210		2.00E-02		820	18	20	170	3.51
Diethylphthalate	1.12	896	8,960		3.50E-03	1.14E-06	142	175	246	222	2.50
Fluorene	1.2	1,690	17		7.10E-04	6.42E-05	7300	12	13	116	4.20
Phenanthrene	0.98	1,000	10		6.80E-04	1.59E-04	14000	14	14	178	4.46
Anthracene	1.28	0.045	0.45		1.95E-04	1.02E-03	14000	0.6	0.6	178	4.45
Di-n-butylphthalate	1.05	13	130		1.00E-05	2.82E-07	170000	2,211	2,212	278	5.60
Fluoranthene	1.25	0.206	2.1		5.00E-06	6.46E-06	38000	7.8	7.9	202	4.90
Pyrene	1.27	0.132	1.3		2.50E-06	5.04E-06	38000	5.0	5.0	202	4.88
Butylbenzylphthalate	1.1	2,900	29		8.60E-06		2430	7.2	7.4	312	4.15

Table C4-1

Summary of Chemical And Physical Properties of Organic Non-Aqueous Phase Liquids Detected in Phase I

Beloit Corporation Blackhawk Facility
Rockton, Illinois

COMPOUND	Density (g/cc)	Water Solubility (mg/L)	1% of Solubility (ug/L)	Ionization Potential (eV)	Vapor Pressure (mm Hg)	Henry's Law Constant (atm-m ³ /mole)	Koc (ml/g)	Soil NAPL Criteria Above WT (mg/kg)	Soil NAPL Criteria Fine-grained (mg/kg)	MW (g/mole)	Log Kow (ml/g)
Benzo(a)anthracene		0.00570	0.06		2.20E-08	1.16E-06	1380000	7.9	7.9	228	5.60
Chrysene	1.27	0.00180	0.02		6.30E-09	1.05E-06	200000	0.36	0.36	228	5.61
bis(2-Ethylhexyl)phthalate	0.98	0.285	2.9		8.60E-06		692	0.21	0.24	391	4.91
Di-n-octylphthalate	0.99	0.285	2.9		8.60E-06		692	0.21	0.24	391	4.91
Benzo(b)fluoranthene		0.0140	0.14		5.00E-07	1.19E-05	550000	7.7	7.7	252	6.06
Benzo(k)fluoranthene		0.00430	0.04		5.10E-07	3.94E-05	550000	2.4	2.4	252	6.06
Benzo(a)pyrene		0.00120	0.012		5.60E-09	1.55E-06	5500000	6.6	6.6	252	6.06
Indeno(1,2,3-cd)pyrene		0.00053	0.005		1.00E-10	6.86E-08	1600000	0.8	0.8	276	6.50
Dibenz(a,h)anthracene		0.00050	0.005		1.00E-10	7.33E-08	3300000	1.7	1.7	278	6.80
Benzo(g,h,i)perylene		0.00070	0.007		1.03E-10	5.34E-08	1600000	1.1	1.1	276	6.51
Heptachlor	1.58	0.180	1.8		3.00E-04	8.19E-04	12000	2.2	2.2	374	4.40
4,4'-DDT		0.00500	0.05		5.50E-06	5.13E-04	243000	1.2	1.2	355	6.19
Methoxychlor	1.41	0.0400	0.40					0.002	0.005		
PCB		0.0310	0.31		7.70E-05	1.07E-03	530000	16	16	328	6.04
Endrin Aldehyde		0.195	2.0		1.78E-07		1700	0.34	0.36	381	

This table presents organic compounds detected during the Phase I RI at the Beloit Corporation - Rockton Facility, and is taken from Table 7-1 of Technical Memorandum No. 1, with the following additions.

A value of one percent of the water solubility is considered to represent the concentration above which a compound may exist as a non-aqueous phase liquid (NAPL). Note that these values have been converted from mg/L to ug/L to facilitate comparison to laboratory data.

Based on the density, the compound is considered to have a density greater than water (DNAPL), or less than water (LNAPL).

Ionization Potentials are taken from the Photovac Technical Bulletin # 11, *Some of the Compounds That can be Detected using the Photovac 10S Series of Portable GC, 1986*. Compounds with a ionization potential less than the PID lamp rating are ionizable by the PID. Note that compounds with a vapor pressure less than water (23.8 mm Hg at 25 degrees Centigrade) and a Henry's Law Constant less than water (1.0E-07) will generally not volatilize sufficiently to be detectable by the PID headspace screening methodology.

Soil NAPL Criteria Above Water table (WT) and Soil NAPL Criteria for fine grained soil represent the solutions to the calculation presented in the text PID SCreening for NAPLs (attached).

PHOTOVAC TECHNICAL BULLETIN NO. 11

SOME OF THE COMPOUNDS THAT CAN BE DETECTED USING THE PHOTOCAC 10S SERIES OF PORTABLE G.C.S

NOTE Many compounds with an ionisation potential of 10.6 eV or less will also be detected by the Photovac TIP* (Total Ionisables Present) Monitor

	eV		eV
Acetaldehyde	10.21	2-bromopropene	10.06
Acetic acid	10.37	3-bromopropene	9.70
Acetone	9.69	2-bromothiophene	8.63
Acetylene	11.41	o-bromotoluene	8.79
Acetylene dichloride	9.80	m-bromotoluene	8.81
Acetylene tetrabromide		p-bromotoluene	8.67
Acrolein	10.10	1,3-butadiene	9.07
Acrylonitrile	10.91	2,3-butadione	9.23
Allene	9.83	n-butanal	9.83
Allyl alcohol	9.67	2-butanal	9.73
Allyl chloride	10.20	n-butane	10.63
Aminoethanol	9.87	1-butanethiol	9.14
Ammonia	10.15	2-butanone	9.53
Aniline	7.70	iso-butanol	10.47
Anisole	8.22	sec-butanol	10.23
Arsine	9.89	tert-butanol	10.25
Benzaldehyde	9.53	2-butanol	10.1
Benzene	9.25	1-butene	9.58
Benzenethiol	8.33	cis-2-butene	9.13
Benzyl chloride	10.16	trans-2-butene	9.13
Benzonitrile	9.71	3-butene nitrile	10.39
Benzotrifluoride	9.68	n-butyl acetate	10.01
Bromobenzene	8.98	sec-butyl acetate	9.91
1-bromobutane	10.13	n-butyl alcohol	10.04
2-bromobutane	9.98	n-butyl amine	8.71
1-bromobutanone	9.54	i-butyl amine	8.70
1-bromo-2-chloroethane	10.63	s-butyl amine	8.70
Bromochloromethane	10.77	t-butyl amine	8.64
Bromodichloromethane		n-butyl benzene	8.69
1-bromo-3-chloropropane		i-butyl benzene	8.68
Bromoethane	10.28	t-butyl benzene	8.68
Bromoethene	9.80	Butyl cellosolve	8.68
Bromoform	10.48	n-butyl mercaptan	9.15
1-bromo-3-hexanone	9.26	i-butyl ethanoate	9.95
Bromomethane	10.53	iso-butyl mercaptan	9.12
Bromomethyl ethyl ether	10.08	i-butyl methanoate	10.46
1-bromo-2-methylpropane	10.09	1-butyne	10.18
2-bromo-2-methylpropane	9.89	2-butyne	9.85
1-bromopentane	10.10	n-butyraldehyde	9.86
1-bromopropane	10.18	Carbon disulfide	10.13
2-bromopropane	10.08	Carbon tetrachloride	11.28
1-bromopropene	9.30	Cellosolve Acetate	

	eV		eV
Chlorobenzene	9.07	2,2-dibromopropane	
Chlorobromomethane		1,2-dichlorobenzene	9.07
1-chloro-2-bromoethane	10.63	1,3-dichlorobenzene	9.12
1-chlorobutane	10.67	1,4-dichlorobenzene	8.94
2-chlorobutane	10.65	1,3-dichlorobutane	
1-chlorobutanone	9.54	1,4-dichlorobutane	
1-chloro-2,3 epoxy propane	10.60	1,4-dichloro-2-butene(cis)	
Chloroethane (ethyl chloride)	10.97	2,2-dichlorobutane	
Chloroethene	10.00	2,3-dichlorobutane	
2-chloroethoxyethene	10.61	3,4-dichlorobutene	
1-chloro-2-fluorobenzene	9.16	Dichlorodifluoromethane(Freon12)	11.75
1-chloro-3-fluorobenzene	9.21	1,1-dichloroethane	11.06
1-chloro-2-fluoroethene(cis)	9.87	1,2-dichloroethane	11.04
1-chloro-2-fluoroethene(trans)	9.87	cis-dichloroethene	9.65
Chloroform	11.37	trans-dichloroethene	9.66
o-chloriodobenzene	8.35	Dichloroethyl ether	
1-chloro-2-methylbenzene	8.72	Dichloromethane	11.35
1-chloro-3-methylbenzene	8.61	1,2-dichloropropane	10.87
1-chloro-4-methylbenzene	8.78	1,3-dichloropropane	10.85
Chloromethylethyl ether	10.08	1,1-dichloropropanone	9.71
Chloromethylmethyl ether	10.25	2,3 dichloropropene	9.82
1 chloro-2-methylpropane	10.66	Dicyclopentadiene	7.74
Chloroprene		Dibutyl amine	7.69
1-chloropropane	10.82	Diethoxymethane	9.70
2-chloropropane	10.78	Diethyl amine	8.01
3-chloropropene	10.04	Diethyl ether	9.53
p-chlorostyrene		N-diethyl formamide	8.89
2-chlorothiophene	8.68	Diethyl Ketone	9.32
o-chlorotoluene	8.83	Diethyl Sulfide	8.43
m-chlorotoluene	8.83	1,2-difluorobenzene	9.31
p-chlorotoluene	8.70	1,4-difluorobenzene	9.15
Cumene (i-propyl benzene)	8.75	Difluorodibromomethane	11.18
Crotonaldehyde	9.73	Difluoromethylbenzene	9.45
Cyanoethene	10.91	1,1-dimethoxyethane	9.65
Cyanogen bromide	10.91	Dimethoxymethane	10.00
3-cyanopropene	10.39	Diiodomethane	9.34
Cyclobutane	10.50	Diisobutyl Ketone	9.04
Cyclohexane	9.98	Diisopropylamine	7.73
Cyclohexanone	9.14	Dimethyl amine	8.24
Cyclohexene	8.95	2,3-dimethylbutadiene	8.72
Cyclo-octatetraene	7.99	2,2-dimethyl butane	10.06
Cyclopentadiene	8.55	2,2-dimethyl butane-3-one	9.18
Cyclopentane	10.52	2,3-dimethyl butane	10.02
Cyclopentanone	9.26	2,3-dimethyl-2-butene	8.30
Cyclopentene	9.01	3,3-dimethyl butanone	9.17
Cyclopropane	10.06	Dimethyl disulfide	8.46
2-decanone	9.40	Dimethyl ether	10.00
1,3-dibromobutane		Dimethylformamide	9.45
1,4-dibromobutane		3,5-dimethyl-4-heptanone	9.04
Dibromochloromethane	10.59	2,2-dimethyl-3-pentanone	8.98
Dibromochloropropane		2,2-dimethyl propane	10.35
1,1-dibromoethane	10.19	Dimethyl sulfide	8.69
Dibromomethane	10.49	Di-n-propyl disulfide	8.27
1,2-dibromopropane	10.26	Di-n-propyl ether	9.27

	eV		eV
Di-i-propyl ether	9.20	4-Heptanone	9.12
Di-n-propyl amine	7.84	1-Hexene	9.46
Di-n-propyl sulfide	8.30	Hexanone	
Epichlorohydrin	10.60	Hexamethylbenzene	7.85
Ethane	11.65	Hydrazine	
Ethanal	10.21	Hydrogen Cyanide	13.91
Ethanol	10.62	Hydrogen selenide	9.88
Ethanethiol (Ethyl mercaptan)	9.29	Hydrogen sulfide	10.46
Ethene (Ethylene)	10.52	Hydrogen telluride	9.14
Ethyl acetate	10.11	Iodine	9.28
Ethyl amine	8.86	Iodobenzene	8.73
Ethyl amyl ketone	9.10	1-iodobutane	9.21
Ethyl benzene	8.76	2-iodobutane	9.09
Ethyl bromide	10.29	Iodoethane (Ethyl iodide)	9.33
Ethyl butyl ketone	9.02	Iodomethane (Methyl iodide)	9.54
Ethyl chloride (Chloroethane)	10.98	1-iodo-2-methylpropane	9.18
Ethyl chloroacetate	10.20	1-iodo-2-methylpropane	9.02
Ethyl ethanoate	10.10	1-iodopentane	9.19
Ethyl disulfide	8.27	1-iodopropane	9.26
Ethylene chlorohydrin	10.90	2-iodopropane	9.17
Ethylene dibromide (EDB)	10.37	o-iodotoluene	8.62
Ethylene glycol dinitrate		m-iodotoluene	8.61
Ethylene oxide	10.56	p-iodotoluene	8.50
Ethyl formate	10.61	Isoamyl acetate	9.90
Ethyl iodide	9.33	Isoamyl alcohol	10.16
Ethyl methanoate	10.61	Isobutane	10.57
Ethyl isothiocyanate	9.14	Isobutyl amine	8.70
Ethyl methyl sulfide	8.55	Isobutyl acetate	9.97
Ethyl propanoate	10.00	Isobutyl Alcohol	10.47
Ethyl trichloroacetate	10.44	Isobutyl formate	10.46
Ethylidene chloride		Isobutyraldehyde	9.74
Ethynylbenzene	8.82	Isopentane	10.32
Mono-Fluorobenzene	9.20	Isoprene	8.85
Mono-Fluoroethene	10.37	Isopropyl acetate	9.99
Mono-Fluoromethanal	11.4	Isopropyl alcohol	10.16
Fluorotribromomethane	10.67	Isopropyl amine	8.72
o-fluorotoluene	8.92	Isopropyl benzene	8.75
m-fluorotoluene	8.92	Isopropyl ether	9.20
p-fluorotoluene	8.79	Isovaleraldehyde	9.71
Freon 11 (CFC13)	11.77	Mesitylene	8.40
Freon 12 (CF2Cl2)	12.91	Mesityl oxide	9.08
Freon 13 (CF3Cl)	12.91	Methanol	10.85
Freon 13 B-1	12.08	Methyl acetate	10.27
Freon 14 (neat)	16.25	Methyl acrylate	10.72
Freon 22 (CHClF2)	12.45	Methyl amine	8.97
Freon 113 (CF3CCl3)	11.78	Methyl bromide	10.53
2-furaldehyde	9.21	2-methyl-1,3-butadiene	8.85
Furan	8.89	2-methylbutanal	9.71
Furfuryl alcohol		2-methylbutane	10.31
Furfural	9.21	2-methyl-1-butene	9.12
Hexachloroethane		3-methyl-1-butene	9.51
n-Hexane	10.18	3-methyl-2-butene	8.67
n-Heptane	10.07	Methyl n-butyl ketone	9.34
2-Heptanone	9.33	Methyl butyrate	10.07

	eV		eV
Methyl cellosolve		(n-perfluoropropyl-iodomethane)	9.96
Methyl Chloroacetate	10.35	(n-perfluoropropyl-methyl ketone)	10.58
Methyl chloride	11.28	Phenol	8.69
Methyl chloroform	11.25	Phenyl ether	8.09
Methylcyclohexane	9.85	Phenyl isocyanate	8.77
4-methylcyclohexene	8.91	Phosphine	9.96
Methylcyclopropane	9.52	Pinene	8.07
Methyl dichloroacetate	10.44	Propadiene	10.19
Methyl ethanoate	10.27	n-propanal	9.95
Methyl ethyl ketone	9.53	Propane	11.07
Methyl ethyl sulfide	8.55	1-propanethiol	9.20
2-methyl furan	8.39	n-propanol	10.51
Methyl iodide	9.54	Propanone	9.69
Methyl isobutyl ketone	9.30	Propenal (Acrolein)	10.10
Methyl isobutyrate	9.98	Propene	9.73
1-methyl-4-isopropylbenzene		Prop-1-ene-2-ol	8.2
Methyl isopropyl ketone	9.32	Prop-2-ene-1-ol	9.67
Methyl methacrylate	9.74	Propionaldehyde	9.98
Methyl methanoate	10.82	n-Propyl acetate	10.04
Methyl mercaptan	9.44	n-Propyl alcohol	10.20
2-methylpentane	10.12	n-Propyl amine	8.78
3-methylpentane	10.08	n-Propyl benzene	8.72
2-methylpropane	10.56	Propylene	9.73
2-methylpropanal	9.74	Propylene dichloride	
2-methyl-2-propanol	9.70	Propylene oxide	10.22
2-methylpropene	9.23	n-Propyl ether	9.27
Methyl n-propyl ketone	9.39	n-Propyl formate	10.54
Methyl styrene	8.35	Propyne	10.36
Morpholine	8.88	Pyridine	9.32
Naphthalene	8.10	Styrene	8.47
Nitric oxide	9.25	Tetrabromoethane	
Nitrobenzene	9.92	Tetrachloroethane	9.32
Nitrotoluene	9.43	1,1,1,2-Tetrachloroethane	
n-Nonane		1,1,2,2-Tetrachloroethane	
5-nonanone	9.10	1,2,3,4-tetrafluorobenzene	9.61
n-Octane		1,2,3,5-tetrafluorobenzene	9.55
3-octanone	9.19	1,2,4,5-tetrafluorobenzene	9.39
4-octanone	9.10	Tetrafluoroethene	10.12
1-octene	9.52	Tetrahydrofuran	9.54
n-Pentane	10.53	Tetrahydropyran	9.26
Pentachloroethane	11.28	1,2,4,5-tetramethylbenzene	8.03
1,3-pentadiene (cis)	8.59	2,2,4,4-tetramethyl-3-pentanone	8.65
1,3-pentadiene (trans)	8.56	1,1,1,2-tetrachloropropane	
Pentafluorobenzene	9.84	1,2,2,3-tetrachloropropane	
Pentamethylbenzene	7.92	Thioethanol	9.29
n-pentanal	9.82	Thiomethanol	9.44
2,4-pentanedione	8.87	Thiophene	8.86
2-pentanone	9.39	1-thiopropanol	9.20
3-pentanone	9.32	Toluene	8.82
1-pentene	9.50	Tribromoethene	9.27
Perchloroethylene	9.32	1,1,1-trichlorobutanone	9.54
Perfluoro-2-butene	11.25	1,1,1-trichloroethane	11.25
Perfluoro-1-heptene	10.48	1,1,2-trichloroethane	
n-perfluoropropyl iodide	10.36	Trichloroethene	9.45

ev

Trichloromethyl ethyl ether	10.08
1,1,2-trichloropropane	9.87
1,2,3-trichloropropane	9.87
Triethylamine	7.50
1,2,4-trifluorobenzene	9.37
1,3,5-trifluorobenzene	9.32
Trifluoroethene	10.14
1,1,1-trifluoro-2-iodoethane	10.10
Trifluoriodomethane	10.40
Trifluoromethylbenzene	9.68
Trifluoromethylcyclohexane	10.46
1,1,1-trifluoropropene	10.9
Trimethyl amine	7.82
1,2,3-trimethylbenzene	8.48
1,2,4-trimethylbenzene	8.27
1,3,5-trimethylbenzene	8.39
2,2,4-trimethyl pentane	9.86
2,2,4-trimethyl-3-pentanone	8.82
n-Valeraldehyde	9.82
Vinyl acetate	9.19
Vinyl benzene (styrene)	8.47
Vinyl bromide	9.80
Vinyl chloride	10.00
4-vinylcyclohexene	8.93
Vinyl ethanoate	9.19
Vinyl fluoride	10.37
Vinyl methyl ether	8.93
o-xylene	8.56
m-xylene	8.56
p-xylene	8.45

D

LABORATORY CUSTODY SOPS

D1 ENSECO
D2 Warzyn
D3 RMT

D1

ENSECO

STANDARD
OPERATING
PROCEDURE

Subject or Title:
SAMPLE RECEIPT AND CHAIN OF CUSTODY

Page 1 of 3

SOP No.:
LP-RMA-0005

Revision No.:
3.0

Effective Date:
1/04/93

Supersedes: 2.0

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1. Purpose:

To document receipt of all samples to the laboratory. To notify lab personnel of all incoming samples. To notify lab personnel of arriving samples that contain short holding parameters. To record the transfer of samples from the client to the lab.

Prepared by:

Date:

January 04, 1993

Management Approval:

Date:

1/6/93

QA Officer Approval:

Date:

1/6/93

SOP No.:
LP-RMA-0005

Revision No.:
3.0

Effective Date:
1/04/93

2. Policies:

Always assign a project number to every group of samples that arrive at the lab regardless of whether work is begun on them or not.

Project numbers are assigned in numerical order, by the LIMS system.

3. Safety:

Proper personal protective equipment must be worn, including lab coats, safety glasses and gloves.

All newly received sample coolers must be opened in an exhaust ventilation hood, and inspected for leaking samples to prevent potential exposure to suspected or unknown hazardous substances that may have vaporized during cooler transport.

If, after initial inspection and during the course of unpacking the samples from the coolers, a situation arises where leaking occurs or any samples begin to offgas or are suspected of offgassing, the sample container and cooler must be returned to the sample receiving hood.

If there are any questions, consult the Enseco Health and Safety Manual or the Health and Safety officer.

4. Procedure:

- a. As samples arrive they are given a unique project number for each group of samples from one client.
- b. For samples arriving by a courier, check that the custody seals are intact. If broken, note this on the sample checklist. (Figure 1)
- c. Sign and date the Chain of Custody (Figure 2). For samples hand delivered, have the client sign and relinquish the chain of custody. Always retain the top copy with the samples and only give a bottom copy to the client.
- d. Open the coolers in a hood, unpack the samples and check the information written on the chain of custody against what was received. Compare the bottles with the Bottle Chain of Custody (Figure 3) and all paperwork received. Check for the correct test, sample matrix and properly preserved bottles for each test requested. Document any discrepancies. Note any discrepancies such as missing samples, broken bottles, pH or cooler temperatures greater than 4°C on the chain of custody form and/or the sample check list. Notify the project administrator of discrepancies so that the client may be contacted in a timely manner.

SOP No.:
LP-RMA-0005

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1/04/93

- e. Fill out the Sample checklist while unpacking the samples. Be certain to fill out both sides of the checklist. Check all sections.
- f. Label all the samples (usually by sampling sites) with a project number and unique sample number (1,2,3,etc.). Record these numbers on the chain of custody next to the client identifications. Have a peer double check that the labelling was done correctly and referenced properly on the chain of custody.
- g. Check the pH of all aqueous non-volatile samples which require pH adjustment for preservation and record on the sample checklist. Use pH strips with a range from 0-14. DO NOT DIP THE STRIPS DIRECTLY INTO THE SAMPLE. Use a disposable micro pipet to extract a small amount of sample and saturate the reactive portion of the pH strip. Compare the pH to the pH indicator colors on the box. Notify the project administrator so that the client may be contacted in a timely manner of any discrepancies (Figure 4).
- h. Look for any inorganic short holding parameters and sign in these samples on the inorganic short holding notification sheet (Figure 5).
- i. Take a picture of the samples. Label a manila file folder with the project number. Place the picture, sample checklist, chain of custody, Level 3 checklist (Figure 6), Bottle Chain of Custody (Figure 3) and any paperwork received in the folder.
- j. Bottles needed to analyze the short holding parameters are hand delivered to the laboratory. Have an analyst sign the short hold record to document receipt of samples.
- k. Place the samples in boxes and store in the walk in cooler on the appropriate team shelves pending log in.
- l. After log-in, deliver the file folder to the appropriate project administrator.
- m. If samples or coolers are labeled with "Quarantine Sample" stickers or other USDA labels, consult the Quarantine Sample SOP for proper handling and storage procedures.

5. Responsibilities:

Sample receiving personnel are responsible for signing the chain of custody upon receipt of samples, for knowing the location of the samples except when used by an analyst, and for signing out maximum security samples. Sample receiving personnel are responsible for noting the short holding parameters only when indicated on the paperwork from the client. Project Administrators must notify sample receiving personnel if others are to be included. Sample receiving personnel are responsible for contacting the project administrator regarding any discrepancies so that the client may be notified in a timely manner.

6. Comments:

For maximum security of samples (beyond the storage in the secured facility) an internal chain of custody is provided. Analysts must sign for the samples in a book and sign them in on return. The samples are stored in one of the locked refrigerators.

FIGURE 1

RMAL Industrial Sample Checklist

Revision 3.0

February 27, 1992

Project #: _____ Date/Time Received: _____

Company Name & Sampling Site: _____

*Cooler #(s): _____ * Place copy of airbill
inside all non-RMAL
Temperatures: _____ coolers. Describe here.
=====

UNPACKING & LABELING CHECK POINTS:

	<u>Y</u>	<u>N</u>	<u>INITIALS</u>
1. Radiation Checked; (record reading if > 15 mr):	_____	_____	_____
2. Cooler seals Intact:	_____	_____	_____
3. Chain of Custody Present:	_____	_____	_____
4. Bottles broken or leaking (comment if Y): -photograph broken bottles-	_____	_____	_____
5. Containers labeled (comment if N):	_____	_____	_____
6. pH of samples taken: -any discrepancies between pH and bottle type? (list below) _____	_____	_____	_____
7. Chain of Custody signed with date, time & lab:	_____	_____	_____
8. CoC agrees with bottle count (comment if N):	_____	_____	_____
9. CoC agrees with labels (comment if N):	_____	_____	_____
10. VOA samples filled completely (comment if N):	_____	_____	_____
11. VOA samples preserved:	_____	_____	_____
12. Sediment present in "D" bottles:	_____	_____	_____
13. Short holding times:	_____	_____	_____
14. Matrix QC verified:	_____	_____	_____
15. Multi phase samples present (comment if Y): -photograph multiphase samples-	_____	_____	_____
16. Clear picture taken & labeled:	_____	_____	_____

Comments: include action taken to resolve discrepancies/problems. Include a hard copy of VAX mail or extra paper if more space is needed.

Sign and Date: _____

FIGURE 1

RMAL Industrial Login Review

Revision 3.0

February 27, 1992

Project #: _____

Dup'd from project #: _____

Set-up By: _____

Dup'd Group Codes: _____

Logged By: _____

Date: _____

SAMPLE CONTROL REVIEW

	<u>Y</u>	<u>N</u>	<u>INITIALS</u>
1. Chain of Custody filled out correctly:	_____	_____	_____
2. Short holding time worksheet correct:	_____	_____	_____
3. Sample bottle/type correct:	_____	_____	_____
4. Overflow sample storage in special instructions:	_____	_____	_____
5. All login paperwork included and correct: Sample list, group code report & acceptance letter	_____	_____	_____
6. Trip blanks, equipment blanks, and field blanks have correct aliquot designation:	_____	_____	_____
7. Sample disc., request list, and acceptance letter in folder:	_____	_____	_____
8. Comments: Include action taken to resolve discrepancies. Include hardcopy of VAX mail, or extra paper, if more space is needed. Sign and date.			

PROJECT ADMINISTRATOR REVIEW:

	<u>Y</u>	<u>N</u>	<u>INITIALS</u>	<u>DATE</u>
Report input sheet:	_____	_____	_____	_____
Invoice information:	_____	_____	_____	_____
All discrepancies resolved:	_____	_____	_____	_____
Sample and test matrices correct:	_____	_____	_____	_____
Sub paper work correct:	_____	_____	_____	_____
Clear picture of sub samples in folder	_____	_____	_____	_____
Special Instructions in LIMS:	_____	_____	_____	_____
Modified component lists checked:	_____	_____	_____	_____
Project due, TAT, received & collected dates OK:	_____	_____	_____	_____
Log released:	_____	_____	_____	_____

CHAIN OF CUSTODY

[illegible]



Rocky Mountain Analytical Laboratory
4955 Yarrow Street
Arvada, CO 80002
303/421-6611 FAX: 303/431-7171

FIGURE 3

BOTTLE ORDER CHAIN OF CUSTODY

ENS-4002

RMAL CLIENT ID		CLIENT P.O. NUMBER		INVOICE NUMBER	
SHIP TO		PICK-UP ON (DATE)	TIME	<input type="checkbox"/> AM	DELIVERED BY (DATE)
				<input type="checkbox"/> PM	
		SHIP BY			
		UPS CHARGES	FED EX CHARGES	CLIENT FED EX ACCOUNT NUMBER	
		COOLERS BILLED TO (SPECIFY CLIENT ID)			

DI _____ GALLONS CARBON FREE _____ GALLONS MILLI-Q _____ GALLONS

NUMBER OF BOTTLES	STANDARD WATER	PARAMETERS	NUMBER OF BOTTLES	BULK WATER	PARAMETERS
	1. 32 oz. poly (WM)	Alkalinity, BOD, Chloride, Color, Res. Chlorine, pH, Chromium (VI), Conductance, Fluoride, Nitrite, MBAS, Ortho-Phos., Solids, Sulfate, Sulfite, Turbidity		20. 1/2 gallon glass	Bulk water analysis
	2. 16 oz. glass (BR) 50% H2SO4	Ammonia, COD, Nitrate, TKN, TON, Nitrate & Nitrite, Total Phos., TOC, Phenolics		21. 1 gallon glass	
	3. 32 oz. glass (BR) 50% H2SO4	TPH, Oil & Grease		SOLIDS	
	4. 16 oz. poly (WM) 20% HNO3	Metals, Hardness		30. 16 oz. glass (WM)	Organics, TPH, Metals, RAD, Oil & Grease
	5. 2-32 oz. poly (BR) 20% HNO3	Gross Alpha, Gross Beta, Uranium, Radium 226, Radium 228		31. 8 oz. glass (WM)	Wet Chem not listed for '30
	6. 8 oz. poly (WM) 50% NaOH	Total and/or Free Cyanide		32. 4 oz. glass (WM)	VOA
	7. 8 oz. poly (WM) Zn Ac & NaOH	Sulfide		TCLP	
	8. 4.5 oz. poly sterilize	Fecal or Total Coliform (use 2 bottles if both required)		33. 32 oz. glass (WM) 4oz. glass (WM)	All other analytes VOA
	10. 3-40 ml glass w/septa. Na2S2O3	THM		OTHER	
	10A. Trip Blank				
	11. 3-40 ml glass w/septa. HCL w/out HCL	VOA, Purgeable Organics			
	11A. Trip Blank				
	12. 2-32 oz. glass (BR)	Base Neutral/Acid Compounds	BLUE ICE REQUIRED <input type="checkbox"/> YES <input type="checkbox"/> NO		
	13. 2-32 oz. glass (BR)	Pesticides, PCBs	SPECIAL REQUIREMENTS		
	14. 32 oz. glass (BR)	Herbicides			
	15. Single: 8 oz. amber glass (BR) Quad: 32 oz. amber glass (BR) 50% H2SO4	TOX-Single -Quad:			
			SAMPLE SAFE/COOLER NUMBERS		

REQUEST BY _____ DATE _____ TIME _____ ☐ AM ☐ PM

RELINQUISHED BY SIGNATURE	RECEIVED BY SIGNATURE	DATE	TIME

FIGURE 4

ENSECO
pH GUIDELINE FOR SAMPLE RECEIPT

BOTTLE TYPE #	EXPECTED pH
1	neutral 5-7
2	<2
3	<2
4	<2
5	<2
6	>12
7	>9
8	neutral 5-7
10	Check done at analysis
10a	Check done at analysis
11	Check done at analysis
11a	Check done at analysis
12	Neutral 5-7
13	Neutral 5-7
14	Neutral 5-7
15	<2

LEVEL 3 CHECKLIST

(To be completed at level 3 review prior to reporting projects.)

CLIENT: _____

PROJECT #: _____

	YES	NO
1. Is the chain of custody complete and properly signed? (CHECK: client IDs, date/time collected, date/time received, sample matrix.)	_____	_____
2a. Have all requested parameters been reported for each sample, including sub-out work and raw data? (CHECK: tests requested and methods referenced.)	_____	_____
b. Have all miscellaneous items been checked? (CHECK: dry weight vs. wet weight, units, "J" values, "B" flags, reporting limits/dilutions, field parameters reported.)	_____	_____
c. Will reanalysis data be reported with original data (if requested)?	_____	_____
3. Are the following forms/checklists available and complete?: -industrial report writing checklists -anomaly forms -out-of-control forms -holding time violation forms	_____ _____ _____ _____	_____ _____ _____ _____
4. Are <u>ALL</u> changes effecting project/program specifications documented and present in the project folder? (This includes phone logs pertinent to the project specifications and project anomalies, all change orders, HT violations, and changes in TAT.)	_____	_____
5. Is the report consistent with the specifications in the Program Assessment Checklist (PAC)? (This includes format, DQOs, etc.)	_____	_____
6. Are the project data consistent with related measurements and parameters, including sub-out work? (Does the data make sense from an historical or site specific perspective?)	_____	_____
7a. Have all non-analytical items and invoice text items been added to the invoice?	_____	_____
b. If the primary deliverable was late, or holding time violations occurred, have penalties been assessed and has the invoice been adjusted (if applicable)?	_____	_____

Comments: _____

PA Initials: _____

Date: _____

STANDARD
OPERATING
PROCEDURE

Subject or Title:

SAMPLE LOG-IN

Page 1 of 3

SOP No.:
LP-RMA-0003

Revision No.:
2.0

Effective Date:
Jan. 04, 1993

Supersedes: 1.0

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1. Purpose:

To create analyses records in the laboratory computer for notification to lab analysts.

2. Policies:

Log-in must be completed within 24 hours of authorization. Authorization occurs after all discrepancies are resolved or in some instances approved addendas are received.

Prepared by:

Date:
January 04, 1993

Management Approval:

Date:

QA Officer Approval:

Date:

1/6/93

1/6/93

SOP No.:
LP-RMA-0003

Revision No.:
2.0

Effective Date:
Jan. 04, 1993

3. Safety:

Always wear gloves, safety glasses and lab coats when handling samples.

4. Procedure:

As log-in proceeds fill out the LOG-IN checklist (Figure 1) and address all the issues on this form.

- 4.1 Once all discrepancies (as described in the sample receipt SOP LP-RMA-0005) have been resolved as best possible, go to the second page of the project screen in LIMS. Make sure that the correct information regarding turn around time, due date, date of receipt are present. Accept the samples by using the sample delivery acceptance soft key. After each soft key, a "do" or saving key must be used to save the changes. Samples cannot be logged in unless this step is performed.
- 4.2 Determine group codes for each sample. Samples with the same analytical requests and same matrix type should be grouped together in one group code. Check with the project administrator regarding any special instructions for the samples.
- 4.3 In LIMS, go to "Group Code" by using the "Group Code" soft key. Add a group code by pressing the "Add Group Code" soft key. In the group code the LIMS sample numbers, matrix type and total sample number fields should be completed.
- 4.4 Next go to "Test List" using the "Test List" soft key. Use the "Add test list" soft key and add the test codes that are applicable to the client request for each sample as listed on the chain of custody and/or other supporting documentation.
- 4.5 Create group codes and add the requested tests for all samples with similar sample matrices and analytical requests.
- 4.6 Add any applicable special instructions for the corresponding groupcode in which the sample(s) is assigned. To input special instructions, the following sequence of soft keys must be used. If others are used, the special instructions will not appear. "Group Code", "Test List", "More Functions", "More Functions", "Group Instruc.", "Add Instruc." If special instructions are long, additional sequences may be necessary. Make sure a note that there are additional sequences is added.

SOP No.:
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- 4.7 Go to the sample list by using the "Sample List" soft key. Add each sample, (use "Add Sample" soft key) filling out all pertinent information such as; sampling date and time, group code, bottle types received. Fill out all fields.
- 4.8 Print the sample list, and a group code report using a control "R" at the sample list screen and group code screen. The paper work should be placed in the project folder. A level 3 checklist (Figure 2) should already be in the the project folder. Abort from this data base and go into "Project reports". Go to acceptance letter and print the acceptance letter by using the appropriate soft key. By using the appropriate soft keys print the sample description and request list paper work. This paper work should also be place into the project folder.
- 4.9 Fill out a chain of custody (Figure 3) for subcontracted work if necessary. A purchase order (Figure 4) must also be filled out. For subcontracting to another Enseco facility fill out the Interlaboratory Analysis Request form (Figure 5).
- 4.10 Perform any compositing, filtering or splitting as necessary. .Create any additional preserved bottles if necessary.
- 4.11 Put samples in the proper locations. Volatiles are placed in separate refrigerators. Waters for organic prep are placed in refrigerators near the Organic prep labs. All other samples are placed by team identification in the walk in refrigerator.
- 4.12 Place project folder in the appropriate location for the team.

5. Responsibilities:

Project Administrator is responsible for reviewing that the log in has been performed correctly based upon the client's requirements and that sample receiving personnel are notified of log in errors. The Project Administrator is responsible for confirming that errors have been corrected. Sample receiving personnel are responsible for transferring the information received with the samples to the laboratory LIMS system and ensuring that a hard copy of this information is placed into the project folder.

6. Definitions:

Special instructions - Typed instructions in LIMS to the teams and analysts that are necessary to complete the work and can not be indicated by using one of the test codes assigned to the samples.

RMAL Industrial Sample Checklist

Revision 3.0

February 27, 1992

Project #: _____ Date/Time Received: _____

Company Name & Sampling Site: _____

*Cooler #(s): _____ * Place copy of airbill
inside all non-RMAL
Temperatures: _____ coolers. Describe here.
=====

UNPACKING & LABELING CHECK POINTS:

	<u>Y</u>	<u>N</u>	<u>INITIALS</u>
1. Radiation Checked; (record reading if > 15 mr):	_____	_____	_____
2. Cooler seals Intact:	_____	_____	_____
3. Chain of Custody Present:	_____	_____	_____
4. Bottles broken or leaking (comment if Y): -photograph broken bottles-	_____	_____	_____
5. Containers labeled (comment if N):	_____	_____	_____
6. pH of samples taken: -any discrepancies between pH and bottle type? (list below) _____	_____	_____	_____
7. Chain of Custody signed with date, time & lab:	_____	_____	_____
8. CoC agrees with bottle count (comment if N):	_____	_____	_____
9. CoC agrees with labels (comment if N):	_____	_____	_____
10. VOA samples filled completely (comment if N):	_____	_____	_____
11. VOA samples preserved:	_____	_____	_____
12. Sediment present in "D" bottles:	_____	_____	_____
13. Short holding times:	_____	_____	_____
14. Matrix QC verified:	_____	_____	_____
15. Multi phase samples present (comment if Y): -photograph multiphase samples-	_____	_____	_____
16. Clear picture taken & labeled:	_____	_____	_____

Comments: include action taken to resolve discrepancies/problems. Include a hard copy of VAX mail or extra paper if more space is needed.

Sign and Date: _____

RMAL Industrial Login Review

Revision 3.0

February 27, 1992

Project #: _____

Dup'd from project #: _____

Set-up By: _____

Dup'd Group Codes: _____

Logged By: _____

Date: _____

SAMPLE CONTROL REVIEW

	<u>Y</u>	<u>N</u>	<u>INITIALS</u>
1. Chain of Custody filled out correctly:	_____	_____	_____
2. Short holding time worksheet correct:	_____	_____	_____
3. Sample bottle/type correct:	_____	_____	_____
4. Overflow sample storage in special instructions:	_____	_____	_____
5. All login paperwork included and correct: Sample list, group code report & acceptance letter	_____	_____	_____
6. Trip blanks, equipment blanks, and field blanks have correct aliquot designation:	_____	_____	_____
7. Sample disc., request list, and acceptance letter in folder:	_____	_____	_____
8. Comments: Include action taken to resolve discrepancies. Include hardcopy of VAX mail, or extra paper, if more space is needed. Sign and date.	_____	_____	_____

PROJECT ADMINISTRATOR REVIEW:

	<u>Y</u>	<u>N</u>	<u>INITIALS</u>	<u>DATE</u>
Report input sheet:	_____	_____	_____	_____
Invoice information:	_____	_____	_____	_____
All discrepancies resolved:	_____	_____	_____	_____
Sample and test matrices correct:	_____	_____	_____	_____
Sub paper work correct:	_____	_____	_____	_____
Clear picture of sub samples in folder	_____	_____	_____	_____
Special Instructions in LIMS:	_____	_____	_____	_____
Modified component lists checked:	_____	_____	_____	_____
Project due, TAT, received & collected dates OK:	_____	_____	_____	_____
Log released:	_____	_____	_____	_____

LEVEL 3 CHECKLIST

(To be completed at level 3 review prior to reporting projects.)

CLIENT: _____

PROJECT #: _____

	YES	NO
1. Is the chain of custody complete and properly signed? (CHECK: client IDs, date/time collected, date/time received, sample matrix.)	_____	_____
2a. Have all requested parameters been reported for each sample, including sub-out work and raw data? (CHECK: tests requested and methods referenced.)	_____	_____
b. Have all miscellaneous items been checked? (CHECK: dry weight vs. wet weight, units, "J" values, "B" flags, reporting limits/dilutions, field parameters reported.)	_____	_____
c. Will reanalysis data be reported with original data (if requested)?	_____	_____
3. Are the following forms/checklists available and complete?: -industrial report writing checklists	_____	_____
-anomaly forms	_____	_____
-out-of-control forms	_____	_____
-holding time violation forms	_____	_____
4. Are <u>ALL</u> changes effecting project/program specifications documented and present in the project folder? (This includes phone logs pertinent to the project specifications and project anomalies, all change orders, HT violations, and changes in TAT.)	_____	_____
5. Is the report consistent with the specifications in the Program Assessment Checklist (PAC)? (This includes format, DQOs, etc.)	_____	_____
6. Are the project data consistent with related measurements and parameters, including sub-out work? (Does the data make sense from an historical or site specific perspective?)	_____	_____
7a. Have all non-analytical items and invoice text items been added to the invoice?	_____	_____
b. If the primary deliverable was late, or holding time violations occurred, have penalties been assessed and has the invoice been adjusted (if applicable)?	_____	_____

Comments: _____

PA Initials: _____

Date: _____

CHAIN OF CUSTODY

ENSECO CLIENT PROJECT SAMPLING COMPANY SAMPLING SITE TEAM LEADER	<table border="1" style="width:100%; border-collapse: collapse;"> <tr> <th colspan="2" style="text-align: center;">SAMPLE SAFE™ CONDITIONS</th> </tr> <tr> <td style="width:70%;">PACKED BY</td> <td style="width:30%;">SEAL NUMBER</td> </tr> <tr> <td>SEAL INTACT UPON RECEIPT BY SAMPLING COMPANY</td> <td>CONDITION OF CONTENTS</td> </tr> <tr> <td>SEALED FOR SHIPPING BY</td> <td>INITIAL CONTENTS TEMP. °C</td> </tr> <tr> <td>SEAL NUMBER</td> <td> SAMPLING STATUS <input type="checkbox"/> Done <input type="checkbox"/> Continuing Until </td> </tr> <tr> <td> SEAL INTACT UPON RECEIPT BY LAB. <input type="checkbox"/> Yes <input type="checkbox"/> No </td> <td> CONTENTS TEMPERATURE UPON RECEIPT BY LAB. °C </td> </tr> </table>	SAMPLE SAFE™ CONDITIONS		PACKED BY	SEAL NUMBER	SEAL INTACT UPON RECEIPT BY SAMPLING COMPANY	CONDITION OF CONTENTS	SEALED FOR SHIPPING BY	INITIAL CONTENTS TEMP. °C	SEAL NUMBER	SAMPLING STATUS <input type="checkbox"/> Done <input type="checkbox"/> Continuing Until	SEAL INTACT UPON RECEIPT BY LAB. <input type="checkbox"/> Yes <input type="checkbox"/> No	CONTENTS TEMPERATURE UPON RECEIPT BY LAB. °C
SAMPLE SAFE™ CONDITIONS													
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SEAL NUMBER	SAMPLING STATUS <input type="checkbox"/> Done <input type="checkbox"/> Continuing Until												
SEAL INTACT UPON RECEIPT BY LAB. <input type="checkbox"/> Yes <input type="checkbox"/> No	CONTENTS TEMPERATURE UPON RECEIPT BY LAB. °C												

DATE	TIME	SAMPLE ID/DESCRIPTION	SAMPLE TYPE	# CONTAINERS	ANALYSIS PARAMETERS	REMARKS

CUSTODY TRANSFERS PRIOR TO SHIPPING				SHIPPING DETAILS	
RELINQUISHED BY (SIGNED)	RECEIVED BY (SIGNED)	DATE	TIME	DELIVERED TO SHIPPER BY	
				METHOD OF SHIPMENT	
				AIRBILL NUMBER	
				RECEIVED FOR LAB	SIGNED
				DATE/TIME	
				ENSECO PROJECT NUMBER	

PURCHASE ORDER

4955 Yarrow Street, Arvada, CO 80002 (303) 421-8611

NUMBER_____

**Show this Purchase Order Number
on all correspondence, invoices,
shipping papers and packages.**

Date _____

To _____

Contact _____

Phone _____

Date Required_____

*Acknowledge promptly if you are unable to ship
complete order by date specified.
Invoice in triplicate.*

Signed by _____

INTERLABORATORY CHAIN OF CUSTODY



PAGE OF

SHIP TO:	ANALYTICAL REQUESTS						SAMPLE CONDITION UPON RECEIPT	SEND RESULTS TO:
ATTENTION:								ATTENTION:
EXPORT ID								COMMENTS
TEST PRICE							WRITTEN RESULTS REQUIRED BY (DATE)	VERBAL/FAC RESULTS REQUIRED BY (DATE)
SUBTOTAL							P.O. No.	
DISCOUNT / SURCHARGE							Q.C. <input type="checkbox"/> STANDARD ENECO <input type="checkbox"/> CLP PROTOCOL <input type="checkbox"/> PROJECT SPECIFIC	
TOTAL							SAMPLE DISPOSAL <input type="checkbox"/> ENECO <input type="checkbox"/> RETURN TO CLIENT <input type="checkbox"/> PHONE	
SPECIAL INSTRUCTIONS							DETECTION LIMITS <input type="checkbox"/> COMMON PRODUCTS <input type="checkbox"/> OTHER	
							HOLDING TIMES <input type="checkbox"/> ENECO <input type="checkbox"/> EPA-CLP <input type="checkbox"/> TIER <input type="checkbox"/> OTHER*	
							RAW DATA COPIES NEEDED <input type="checkbox"/> YES <input type="checkbox"/> NO	
							CUSTODY SEALS INTACT <input type="checkbox"/> YES <input type="checkbox"/> NO <input type="checkbox"/> WET WEIGHT <input type="checkbox"/> DRY WEIGHT	
							RELINQUISHED	DATE / TIME
							RECEIVED	DATE / TIME

D2

WARZYN

CHAIN-OF-CUSTODY, LOG-IN AND TRACKING PROCEDURES FOR GEOTECHNICAL SAMPLES

Scope and Application:

This procedure is applicable to the log-in of samples and addresses such areas as: sample custody, log-in, labeling and preservation of samples. This procedure begins as the samples enter the laboratory and follows the samples to their destruction.

Procedure:

1. **Chain-of-custody Procedure:** Samples arriving in the laboratory are accompanied by some type of custody record. The most common type of record is that shown in Figure 1. Follow the instructions below for all levels of chain-of-custody.

Superfund Level Chain-of-custody:

- a. Refer to the "Chain-of-custody - Superfund Level" SOP.

Routine Chain-of-custody:

- a. Samples arriving in the laboratory are accompanied by a chain-of-custody record (refer to Figure 1).
- b. Review the custody form with the samples received and see that the spaces marked #1 - #12 have been accurately completed by the sampler.
- c. Any discrepancies with the chain-of-custody record or the samples should be immediately brought to the attention of the customer service relations person so that the problems can be solved in an efficient manner.
- d. If samples were shipped, note the courier name in the remarks section (#15) of the chain-of-custody record.
- e. Record any other comments such as: temperature upon receipt, preservation (if done in lab), problems, etc, in the remarks section (#15).
- f. Fill in the lab numbers in the section marked #16 (refer to the log-in portion of this procedure for how to obtain lab numbers).
- g. Sign and date the chain-of-custody record (#13 - #14).

- h. Route the custody record to the data management office when log-in is complete.

Note: The laboratory is a secured area with strict limited access. Any samples arriving to the laboratory during "off hours" are stored in the laboratory refrigerator or storage area until they are logged in. Laboratory personnel are responsible for the care and custody of the samples during the analysis stage and until the samples are removed from the refrigerator or storage area and properly disposed of.

Construction Samples

- a. Samples arriving in the geotechnical laboratory are accompanied by the blue "Geotechnical Laboratory Set-up Sheet" (refer to Figure 3).
- b. Review the Set-up Sheet with the samples received to see that Set-up Sheet has been accurately completed.
- c. Any discrepancies between the Set-up Sheet and the samples should be brought to the immediate attention of the lead project personnel so that problems can be solved in an efficient manner.
- d. Date the Set-up Sheet for when it is received in the laboratory.

2. Log-In Procedure

- a. **Sample Information:** Document the condition of the samples upon receipt. Note any irregularities or problems on the chain of custody or the laboratory Set-up Sheet.
- b. **Numbering Samples:** Number the samples on the chain-of-custody form. Record laboratory numbers used in the black laboratory book. For construction samples assign laboratory numbers on the laboratory set-up sheet.
- c. **Subcontracted Work Required:** Refer to SOP for further instructions on subcontracting samples.
- d. **Special Accounting Notes:** If there is a discount or special proposal for sample analysis to be charged, record this information on chain-of-custody and laboratory Set-up Sheet.
- e. **Labels:** Check that all samples are labeled with; project number, sample number, date, and location

Computer Log-In:

1. Analysis requests are entered into the Laboratory Information Management System (LIMS). The LIMS database is used for scheduling of analytical work and to produce accounting invoices. Analysis requests are entered into the LIMS database as follows:

Computer Log-on

- Turn on the computer terminal.
 - Enter user name > last name [return].
 - Local prompt > c_chico [return].
 - User name: ACSORA [return].
 - Password: [return].
 - Please enter your last (15 characters max.): Last name: [return].
 - \$Labmenu [return].
 - Username: Last name [return].
 - Password: [return].
 - Select Log-in/Order entry and press [commit].
2. The log in screen will appear as shown in figure 2. Using information on the chain-of-custody, proceeds as follows:
 - a. Enter project number (return). Account number and name will be filled in automatically. If the information matches the project name on the chain-of-custody, press return.
 - b. Sample date automatically defaults to yesterday's date. If this is correct, press return or override with correct date.
 - c. Enter sample identification from **station location** on the chain-of-custody and press return.
 - d. Enter proper matrix number and press return.

- e. **Desc:** This is used if sample identification is longer than the number of characters in the sample identification field. (Return to override or enter information and return.)
- f. Date received automatically defaults to today's date. If correct, press return or override with correct date.
- g. Days to process automatically defaults to 14 days. If this is correct press return or for standard turnaround time (usually 4 weeks) or if rush work, (indicated on request form) override with the number of days until due.
- h. **Duedate:** First date listed is report date, second date is the lab due date which can be changed if necessary by changing days to process.
- i. **Comments:** Enter special tests or other information needed to analyze samples correctly and press return.
- J. **Manger:** Filled in automatically; press return if correct or override.
- k. **Markup/Discount:** Return if correct or override.
 - **% Markup/Discount:** For accounting. Enter a negative number for a discount (i.e. -15 for 15%) and a positive number for markups (i.e. 100 for 2 x price). Check accounting sheets for projects with special pricing.
- l. To enter parameters, press **Next Screen** (see Figure 2A).

Note: To move from block to block press Next/Previous screen. To move from field to field, press Return/Tab or F12 (previous field) key.
- m. Matrix automatically defaults to matrix entered above (return or override).
- n. Enter the proper code name for the test to be run from report MF01. Use parent codes if applicable for quicker entry. (Press the down arrow key, return.) To delete use remove key.
- o. To enter address for the report, press **Next Screen** (see Figure 2B).
- p. **Type:** Report (return).
- q. If an address has already been entered and matches the chain-of-custody press **Commit** and return.

- r. If no address appears, press return until cursor is in the City field. Type in the city and press return.
 - s. Type in state (press **Commit** and return). After city and state have been entered for the first sample, it will automatically be copied for the rest of the sample numbers in a single log-in.
3. To duplicate the entry for another sample on the same chain-of-custody:
 - a. If next sample to be entered has all the same information as the previous sample entered, press **Duplicate Record** key (F7).
 - b. Enter sample identification and press **Commit** key, return.
 4. To duplicate only the information in the upper block of the log-in screen (sample date, matrix etc.):
 - a. Press **Create Record** key (Insert Here) and enter sample identification.
 - b. Press **Previous Screen** (F12) if sample date needs to be changed.
 - c. Press **Next Screen**, return. Then press **Next Screen** again to enter new product codes for the analyses requested.
 - d. Press **Previous Screen** to enter new sample or to quit.
 - e. Press **Clear Form** (F17) to enter a new chain-of-custody (new log in number).
 - f. Press **Exit/Cancel** (PF4) to exit from the log-in screen.
 - Press **Exit/Cancel** to exit from the LIMS system.
 - At the VMS \$ prompt, enter LO (return) to log off the VAX computer.
 - Enter N (return).
 5. If in Step 2a. the computer says project number is invalid:
 - a. Press **Exit/Cancel** (PF4).
 - b. Enter "Y" (return).
 - c. On the LIMS Main menu, use the up arrow key to position the cursor on "Seedpak Management". Press **Commit** key.
 - d. Use the down arrow key to move to project entry. Press **Commit** key.

- e. Enter project number (return).
 - f. Enter account number (9999) and return.
 - g. Enter description for chain-of-custody (project name).
 - h. Enter manager from chain-of-custody.
 - i. Press **Exit/Cancel** (PF4), twice.
 - j. At the LIMS main menu, select log-in and press **Commit**.
6. To print daily log-in reports:
- a. Print out the appropriate FS02 reports for only the samples logged in that day. The computer printout must be checked against the custody record or field sheets by the inorganic/organic supervisors so that any errors can be corrected. Print a report as follows:
 - b. Select "Seedpak Reporting" from the main menu, and press **Commit**.
 - c. Select FS02 and press **Commit**.
 - d. Enter today's date, press **Commit** and return.
 - e. The inorganic/organic supervisors are responsible for updating and printing lab schedules as they require them.
 - f. Any "rush" work is to be brought to the inorganic/organic supervisors' attention immediately.

3. Sample Storage

- a. All samples will be stored in the temporary storage area or moisture room until completion of laboratory work.
- b. Upon completion of work samples will be transferred to long-term warm storage.
 - 1. Chain-of-custody samples from analytical laboratory will be returned to analytical laboratory.
 - 2. Construction samples will be stored at the geotechnical laboratory.
- c. All test designated samples that remain after testing will be resealed and stored.

- d. All sample storage areas will be kept locked and are restricted to laboratory personnel only.
- e. Any sample removed from storage must be put back in the original location.

4. Sample Security

- 1. Samples will be stored in a secure area.
- 2. Access to the laboratory will be through a monitored area. Other outside-access doors to the laboratory will be kept locked.
- 3. Visitors will sign a visitors log (located at the reception area and will be escorted while in the laboratory area.
- 4. Refrigerators, freezers, and other sample storage areas are secure as they are located within the laboratory.
- 5. Only laboratory personnel have access to the laboratory.
- 6. Samples will remain in secure sample storage until removed for sample preparation or analysis.
- 7. After a sample has been obtained from storage by the analyst, the analyst is responsible for the custody of the samples. Each analyst must return the samples to the storage area before the end of the working day.

Sample Disposal:

Due to limited storage facilities available, samples will be stored for three months after report issue date. Sample storage time may be extended upon client's and/or Project Manager's request for an additional 3 month period storage fee.

D3

RMT

STANDARD OPERATING PROCEDURE

PROCEDURE TITLE: Receiving, Log-in, Storage, Analysis and Disposal of Samples

DEPARTMENT: All Departments

PROCEDURE:

Scope

The following steps are taken by the Laboratory Personnel when samples arrive in the laboratory for analysis:

- Case seals and sample seals are examined for integrity.
- Samples are examined for proper labels, damage, proper preservatives and temperature, and referenced to the chain-of-custody.
- The chain-of-custody form (Figure 1.07-1) is signed.
- Samples are stored under the proper environmental conditions, and their location and condition are noted in the appropriate log.
- Samples are entered into the Laboratory LIMS System.
- A Sample Acknowledgment Form is filled out and sent to the project manager.
- Supervisors are notified of the arrival of the samples.
- Internal chain of custody form is completed and samples are stored in a secure area.
- Work lists and work schedules are prepared for sample analysis.
- Both the preparation and analysis of samples will be documented.
- Samples are returned or disposed of after analysis is completed.

Receipt

The company mail clerk, or when unavailable, the sample custodian or his/her designated backup will sign for the receipt of samples from the commercial carrier:

Log-in

Samples should be logged in as soon as possible after receipt. If samples arrive during non-business hours, they must be logged in immediately the next business day. The date of receipt should be the date that the form was completed, and the actual receipt date should be noted in the "comments" section on the custody form.

Before opening each cooler, check for the presence/absence of custody seals or custody tape on each cooler, and record this information in the "Seal # _____ recvd. intact by" box on the RMT Chain-of-Custody form.

Open each cooler and remove the enclosed documents. Note the presence/absence of the Chain-of-Custody, RMT workorder forms, and air bills or bills of lading. These will be noted on the Chain-of-Custody in the "Comments" section or a separate piece of paper if the Chain-of-Custody is absent.

Sign the RMT Chain-of-Custody form in the last box "received by;" note date/time of receipt. Write the air bill tracking number and shipper name on the previous "relinquished by" box providing the air bill is in agreement with the one documented by the shipper. Any discrepancies must be noted. Attach the air bill to the Chain-of-Custody.

Remove all sample containers from the cooler and place them on the log-in counter. Items to check and note condition or presence/absence on the custody form are:

- The condition (intact or broken) of all samples and their respective custody seals, if present.
- The representative temperature of each cooler, using an unpreserved water sample, or in the case of soils or samples with no headspace, the presence of ice sufficient to cool the samples to $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ as per standard RMT procedure.
- The disposal of any damaged samples will be in an appropriate manner and will be documented.
- The pH of each preserved sample and the notation of any preservative added in the designated box on the Chain-of-Custody form.

- Presence/absence of sample tags - For RMT procedures these are usually non-detachable labels affixed to the containers. This should be noted in the "Comments" section of the Custody form.
- Compare the sample tag ID's on the sample to the ID's on the Chain-of-Custody in the space labeled "Sample Station ID."
- RMT workorder forms specifying which analysis is requested on each sample should also accompany the shipment of samples. The presence of this form serves as a check to ensure that the correct bottles for each requested analysis have been received.

LIMS entry

The sample custodian will assign consecutive laboratory sample numbers to each sample received. Laboratory sample numbers will be listed on the Chain-of-Custody to cross reference the sample tag ID numbers. The laboratory sample numbers will be written on each sample bottle on the affixed label or sample tag.

Initiate a Sample Receipt Acknowledgment form after the samples been entered into the LIMS system. Attach the Sample Receipt Acknowledgment form to the Chain-of Custody form and the work order.

Storage

The location of each type of sample bottle will be noted in the appropriate grid space in the RMT sample log/refrigerator tracking book (Figure 1.07-2).

The storage area will be kept secure at all times. Each sample will be logged into the appropriate refrigerator or bin. The sample custodian will initial and date the separate Refrigerator Tracking Log (Figure 1.07-3) or Bin Log (Figure 1.07-4) sheet kept next to each refrigerator or bin after storing each sample. Each subsequent analyst will sign and date the refrigerator or bin log when removing or replacing samples. All transfers will be documented on an internal chain of custody record (refrigerator or bin log). After a sample has been removed from storage by the analyst, the analyst is responsible for the custody of the sample. Each analyst must return the samples to the storage area before the end of the working day or prior to the end of his/her shift. The refrigerators are maintained according to RMT written procedure.

Samples and extracts will be stored after completion of analysis in the proper storage area. Volatile samples will be stored separately from other samples. Standards will not be stored with samples.

Laboratory Security

Access to the laboratory will be through a monitored area. All laboratory personnel are issued a security card which permits entry to the laboratory. Other outside access doors to the laboratory are kept locked. Visitors will sign a visitors log and be escorted while in the laboratory. Samples will remain in secure sample storage until removed for sample preparation or analysis.

Raw Data

All hand written raw data pertaining either directly or indirectly to a sample analysis must be written in water insoluble black ink. All raw data should be on the appropriate documents (e.g., laboratory notebook, bench sheet, calibration logs, etc.); data must not be recorded on "scratch paper" and then transferred to the appropriate documents. If for any reason data are not recorded on the appropriate documents the paper containing the original data should be, if possible, permanently attached to the document. If this is not possible, the paper should be referenced on the document. Stray marks and numbers on documents are not acceptable; all data must be legible and identified. Columns of data should be labeled, and the units should be defined (i.e., peak area, concentration, absorbance, weight, etc.).

Bench Sheets and Laboratory Notebooks

Any employee collecting raw data must sign and date the data in water insoluble black ink and describe the kind of procedure conducted (e.g., sample extraction, digestion, analysis, etc.). Computer generated data must be signed and dated (if not initialed and dated on the computer printout) by the responsible individual operating that instrument.

It is essential that any unusual occurrences or data that may affect the analysis be recorded and documented in its proper place.

Any spillage, contamination, quality control failure, or other reason that necessitates that a sample be rerun should be documented, and a short explanation should be given.

- Unique instrument identification for each instrument used during the collection of raw data should be included on the bench sheet or in the laboratory notebook.

Instruments

All instruments must have an instrument standard operating procedure (SOP), analytical run log, maintenance and calibration log.

Each instrument must be calibrated according to the procedure and the frequency described in the instrument SOP, analytical method or the Quality Assurance (QA) Manual, and the calibration recorded in the log and the samples analyzed. Dates and explanations of routine and non-routine repairs must also be recorded in the maintenance log. Records of non-routine maintenance should document the nature of the defect, how and when the defect was discovered, and any remedial action taken in response to the defect.

Analytical Methods and Procedures

If an analytical method or procedure is modified or if an alternative procedure is used, which is different from the analytical method currently used in the laboratory, the actual method or procedure used should be written on or attached to the raw data. These variations should be justified and approved in writing by the section supervisor. Special requests by clients should be documented in the raw data.

Data Handling and Calculations

All looseleaf data must contain a project number or other identification to designate it as part of a particular project or sample.

All computer calculation sheets must be attached to a bench sheet or stored in an orderly fashion, being cross-referenced in some manner. All manual calculations should be recorded on the bench sheets. If calculation sheets are used, they shall be attached to the bench sheets, or cross-referenced to the bench sheet. All chromatograms and instrument printouts should be labeled with instrument parameters.

Each chromatogram injection should be identified by sample number (or Standard), injection volume, and must be signed and dated by the person responsible for injection. If a sample is auto-

injected, one signature and date are adequate for the group of samples in that run.

Each AA, ICP, Lachat, or other appropriate instrument printout should be identified with sample number (or standard) and must be signed and dated by the person responsible for each analysis. If a sample is auto-injected, one signature and date are adequate for the group of samples in that run.

Standard Curves and Standard Preparation

Record the data for the standard curve on the bench sheet with appropriate units. In those cases where a standard curve is applicable to more than one bench sheet, record on each bench sheet the location of the standard curve data.

Dilutions from stock standards should be labeled with the date the dilution was made and the date that the stock standard was prepared. These dates should be recorded on the bench sheet along with the standard curve data.

Reporting an Assay

It is the analyst's responsibility to review the raw data and the bench sheet prior to being reviewed by a second analyst. The second analyst reviews the data for calculations and data completeness. Upon reviewing the raw data the second analyst signs and dates the bench sheet. The section supervisor reviews the data for quality control (QC) criteria (calibration, frequency of QC samples and QC results). The section supervisor approves the data for LIMS entry by initialing and dating the raw data. The section supervisor reviews the LIMS generated report for technical significance. The report then goes to the laboratory director for final approval. The laboratory director reviews the final report. Upon approval the laboratory director signs and dates the final report. The QC coordinator reviews approximately 10% of the final reports. This is done prior to the review and approval of the report by the laboratory director. The QC coordinator reviews the report for QC criteria, completeness, QA compliance and traceability. Please see the attached flow chart for the process of reviewing raw data in the laboratory.

Sample Disposal

Samples may be completely consumed during analysis, returned to the client or sampling location, stored under required environmental conditions if reanalysis is anticipated or under

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ambient conditions if reanalysis is not likely, or disposed by the laboratory. Samples and extracts shall usually be disposed within sixty days unless otherwise specified.

The Laboratory Director, or his/her designee, shall determine the method and time for disposal if not specified by the Project Manager.

Some waste may be disposed of in a sanitary sewer as permitted by 40 CFR 261.3(a)(2)(iv). Some samples may be hazardous because of their general characteristics or because they are listed in 40 CFR Part 261. shipping of these materials is addressed in 40 CFR 172.02, 172.03, 172.04, 172.300 and 172.400.

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DATE: APRIL 1990



Madison, WI 53717
744 Heartland Trail
Phone (608)831-4444
FAX (608)831-3334

Washington, DC

Santa Monica, CA

Grand Lodge, MI

Greenville, SC

F-268 (R2/88)
(Use Black Ink Only)

CHAIN OF CUSTODY RECORD

Sample Type: (GW, WW, SW, Soil, Other)

No 009806

Bottles Prepared by: _____ Date/Time _____ Office Code: _____
(State)

Project No. _____ Client: _____

RMT Lab NO. _____ Yr. _____ Date _____ Time _____ Sample Station ID _____

Total Number
Of Containers

Container Inventory

Filtered (Yes/No) _____
Preserved (Code) _____
Refrigerated (Yes/No) _____

Code: A - None
B - HNO₃
C - H₂SO₄
D - NaOH
E - _____

Comments: _____

FIGURE 1.07-1

SAMPLER Relinquished by (Sig.) ①		Date/Time	Received by (Sig.) ② Shipper Name & #		Date/Time	HAZARDS ASSOCIATED WITH SAMPLES (For Lab Use Only) Receipt Temp _____ Receipt pH _____ Client P.O. Number _____ Subsequent Analysis: _____ (Check)				
Relinquished by (Sig.) ③		Date/Time	Received by (Sig.) ④ Shipper Name & #		Date/Time					
Relinquished by (Sig.) ⑤		Date/Time	Received by (Sig.) ⑥ Shipper Name & #		Date/Time					
Seal #	at'chd by	<input type="radio"/>	Recvd. Intact by	<input type="radio"/>	Seal #	at'chd by	<input type="radio"/>	Recvd. Intact by	<input type="radio"/>	Date Resubmitted

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FIGURE 1.07-4

LABORATORY SOLID WASTE INVENTORY

Bin #:

Date	Project	Project Number	Sample Numbers	Project Manager	Container Type	Checked In By (Date/Initial)	Checked Out By	Checked In By	Checked Out By	Checked In By	Checked Out By	Checked In By
						1.	2.	3.	4.	5.	6.	7.
						1.	2.	3.	4.	5.	6.	7.
						1.	2.	3.	4.	5.	6.	7.
						1.	2.	3.	4.	5.	6.	7.
						1.	2.	3.	4.	5.	6.	7.
						1.	2.	3.	4.	5.	6.	7.
						1.	2.	3.	4.	5.	6.	7.
						1.	2.	3.	4.	5.	6.	7.
						1.	2.	3.	4.	5.	6.	7.
						1.	2.	3.	4.	5.	6.	7.
						1.	2.	3.	4.	5.	6.	7.
						1.	2.	3.	4.	5.	6.	7.
						1.	2.	3.	4.	5.	6.	7.
						1.	2.	3.	4.	5.	6.	7.
						1.	2.	3.	4.	5.	6.	7.
						1.	2.	3.	4.	5.	6.	7.
						1.	2.	3.	4.	5.	6.	7.

(96.04:LAB:LF-388)

14.0 QUALITY ASSURANCE/QUALITY CONTROL AUDITS

Audits of the RMT Laboratory are conducted for several reasons:

- . To identify potential or actual deficiencies so that the problems may be corrected.
- . To assure that RMT procedures and methods are being followed.
- . To determine that records are properly filled out and filed.
- . To assure that regulatory requirements are met.
- . To establish that quality assurance objectives are met.

Audits are not conducted to assign blame.

14.1 LABORATORY AUDITS

Monthly, the QC Coordinator, shall conduct an in-depth audit of the RMT Laboratory. This audit shall include the following items:

- . Sample maintenance
 - Are stated temperatures for sample storage provided?
 - Are samples processed and tested within prescribed holding times?
 - Are samples properly logged in?
- . Calibration
 - Are calibrations performed as required?
 - Are they properly documented in instrument log books, or as part of project data if required?
 - Do calibration results indicate a trend in instrument performance?
- . Preventive maintenance
 - Are adequate spare parts available?
 - Do specific instruments have repeated maintenance problems?
 - Is preventive maintenance performed and properly documented?

- . Receipt and storage of standards, chemicals, and gases
 - Are all reagents, chemicals, and gases purchased for use in the laboratory of adequate grade for the intended use?
 - Are certifications of material compositions provided when required?
 - Are materials adequately stored to prevent degradation?
 - Are materials kept beyond stated shelf life?
 - Are internal standards properly prepared and stored?
 - Are internal standards kept beyond stated shelf life?
- . Analytical Methods
 - Are the methods used appropriate for project requirements?
 - Are alternate methods approved for use?
- . Data Verification
 - Are data processed and validated as prescribed?
- . Records Management
 - Are the records of analyses complete and properly identified?
 - Are documents submitted to the record system in a timely manner and are they properly maintained?

The following areas shall be audited:

- . Sample Entry
- . Inorganic Laboratory
- . Organic Laboratory
- . Report Production

The laboratory audit shall consist of a general audit and a specific method/procedure audit.

- . A general audit shall be an overview of the whole laboratory from sample receipt to sample disposal for compliance with the Quality Assurance Manual (Figure 14-1).
- . A specific method/procedure audit shall be a detailed in-depth review of an actual method or procedure. This may include

sample receipt (Figure 14-2), standard/reagent/solution preparation (Figure 14-3), sample preparation/extraction (Figure 14-4), sample analysis (Figure 14-5) or data verification (Figure 14-6).

After the general and/or specific audits have been conducted, the Quality Assurance Coordinator shall complete the Laboratory Audit Record form (Figure 14-7). Any problems, observations, and findings which are identified by the Quality Assurance Coordinator shall be discussed with the Group Supervisors.

A written report (Figure 14-8) summarizing the findings of the audit shall be sent to the Group Supervisors who shall be responsible for corrective action. The Group Supervisor, or the employee designated to respond to the report, shall sign and date the report.

The original copy of the audit report form with responses shall be routed to the Laboratory Director for review and comments. The Laboratory Director shall sign and date all audit response forms to document that they have been reviewed. The audit reports forms shall be sent to the Quality Assurance Coordinator for filing.

Deficiencies reported as a result of participation in round-robin studies or outside audits shall be handled in the same manner.

E

**DATA VALIDATION PROCEDURE FOR
EVALUATING INORGANIC DATA**

DATA VALIDATION PROCEDURE FOR EVALUATING INORGANIC DATA

Scope and Application:

This standard operating procedure (SOP) describes Warzyn's procedure for evaluating inorganic data according to procedures specified in a Quality Assurance Project Plan (QAPP). Each analytical procedure is specific to a project and is found or referenced in the QAPP. The reviewer must evaluate whether the procedure was adhered to and that the required Quality Control (QC) requirements were met as described in the QAPP and evaluate the usability of the data.

References:

1. Laboratory Data Validation Functional Guidelines for Evaluating Inorganics Analyses, Hazardous Site Evaluation Division, U.S. EPA, July, 1988 (or most current).
2. Statement of Work for Inorganics Analysis, U.S. EPA Contract Laboratory Program, SOW No. 788, July, 1988 (or most current).
3. Quality Assurance Project Plan (QAPP) containing the analytical procedures required for evaluation. Each QAPP is specific to a project.

Requirements:

1. It is recommended that the reviewer be familiar with the RAS Inorganics validation process (refer to the Validation Guidelines and Statement of Work above) prior to evaluating this data. The evaluation and action criteria described in this SOP are very similar to the RAS Inorganics validation process.
2. All completed validated data packages must include the following:
 - a. Data Validation Narrative
 - b. Validated Results; with data qualifiers added and pages stamped "VALIDATED".

NOTE: It is not necessary to include the raw data with the completed validated data package, since a copy of the raw data package already exists in the project file. Only include raw data if there are major concerns or deficiencies with the data set.

Procedure:

1. Screen the data package to see that the samples and analytes undergoing evaluation concur with what was requested on the "Request for Data Validation" form (refer to Figure 1). If discrepancies occur, contact the data management coordinator for clarification prior to continuing.

2. Review the QAPP and the specific analytical procedure for the analyte undergoing evaluation. They will contain:
 - a. A summary of the method required and method reference,
 - b. The applicable matrices and recommended hold times,
 - c. An approximate number of samples and matrix,
 - d. Special technical instructions,
 - e. Required deliverables,
 - f. Data Requirements (Detection Limits, Precision, etc.), and
 - g. Quality Control Requirements.
3. The reviewer must evaluate the data according to the quality control criteria specified in the QAPP. The evaluation of each analyte, in most cases, will include a review of the following criteria:
 - a. Methodology,
 - b. Raw Data/Quality Control Deliverables,
 - c. Detection Limits,
 - d. Holding Times,
 - e. Calibration (Initial and Continuing),
 - f. Blanks (Calibration and Preparation),
 - g. Laboratory Control Samples (EPA Reference Samples),
 - h. Laboratory Duplicates,
 - i. Matrix Spikes,
 - j. Sample Result Verification,
 - k. Field Duplicates,
 - l. Other (as specified in the procedure),
 - m. Overall Assessment for a Case.

4. Evaluate the data for the criteria above as follows:

A. METHODOLOGY

Evaluation

1. Examine the data to determine if the acceptable analytical methodology was used as indicated in the QAPP.
2. Determine if the analytical procedure was followed.

Action

1. If an acceptable analytical method was not used, contact the Project Manager. It is the Project Manager's responsibility to inform the reviewer to continue the validation process of the data as received, or to reject the data. In some cases reanalysis or

resampling may be required, however the decision is left up to the Project Manager. The Project Manager may require that the reviewer determine the "usability" of the data package as submitted prior to deciding what formal action should be taken.

2. If the analytical procedure was not followed as stated, use professional judgement in determining whether the data should be qualified as estimated or unusable.

B. RAW DATA/QUALITY CONTROL DELIVERABLES

Evaluation

1. The QAPP will specify what information should be submitted as part of the data package. Determine whether the data package submitted is complete as described in the QAPP. Usually the following will be required:
 - Method used,
 - Calibration/standardization information,
 - Bench records tabulating the order of analysis of samples, standards, blanks, duplicates and spikes with the resulting instrument readouts and final concentrations. Records submitted should be complete enough such that all results could be recalculated from this raw data.
 - Photocopy of all instrument readouts (strip charts, printer tapes, etc.)
 - Reference (QC) samples to be identified by source, lot number and sample number, with corresponding true values and 95% confidence limits provided.
 - Custody records

Action

1. If the data package is not complete, contact the data management coordinator. The data management coordinator will contact the appropriate laboratory or project manager and obtain the information necessary to complete the evaluation process.

C. DETECTION LIMITS

Evaluation

1. Evaluate whether the detection limits required in the QAPP were achieved by the method used.

Action

1. If detection limits were not reported at least to what was required in the QAPP, review the raw data to determine if the detection limits were achievable. If they were, recalculate the data and report results to the required detection limits.
2. If detection limits were not achievable by the laboratory, determine why (laboratory sensitivity problem or detection limits unobtainable for the method used. Inform the project manager of

the situation. The project manager will determine whether the evaluation should be completed.

D. HOLDING TIMES

Evaluation

1. Determine whether the established holding times were met. The holding time is established by comparing the DATE SAMPLED with the DATE OF ANALYSIS found on the raw data. Required hold times should be noted in the QAPP.
2. Examine the digestion/distillation logs to determine if samples were preserved at the proper pH.

Action

1. If hold times were not noted in the QAPP, use the EPA-recommended hold times and apply these hold times to both water and soil samples.
2. If holding times and preservation criteria are not met, qualify all results > Instrument Detection Limit (IDL) and estimated (J) and results < IDL as estimated (UJ).
3. Use professional judgement in cases where the holding time is grossly exceeded. The expected bias would be low and the reviewer may determine that results < IDL are unusable (R).

E. CALIBRATION

Evaluation

1. Verify that the instrument was calibrated daily and each time the instrument was set up using the correct number of standards and blank. In cases where a curve set-up is not applicable (i.e. a titrimetric procedure), verify that the titrants were standardized as required in the procedure.
2. Verify that the correlation coefficient is ≥ 0.995 (unless otherwise specified).
3. Verify that the calibration verification standards (ICV/CCV) were run at the appropriate frequency and that results were within acceptable limits as stated in the QAPP.
4. Recalculate approximately 10% of the ICV/CCV using the following equation:

$$\%R = \frac{\text{Found}}{\text{True}} \times 100$$

Due to possible rounding discrepancies, allow results to fall within 1% of the contract windows.

Action

1. If the minimum number of standards as required in the QAPP were not used for initial calibration, or if the instrument was not calibrated daily and each time the instrument was set up, qualify the data as unusable (R).
2. If the correlation coefficient is < 0.995 (or as specified in the QAPP), qualify results $> IDL$ as estimated (J) and results $> IDL$ as estimated (UJ).
3. If standardization of titrants was required and not performed, use professional judgement in qualifying the data. The reviewer should review ICV/CCV, EPA reference sample results and other criteria prior to determining if the data should be qualified as estimated (J if $> IDL$; UJ if $< IDL$) or unusable (R).
4. If the ICV/CCV %R falls outside the acceptable limits, use professional judgement to qualify associated samples. If possible, indicate the bias in the review. The following guidelines may assist the reviewer in qualifying the data:
 - If the ICV/CCV %R is significantly greater than the upper control limit (UCL), qualify associated results $> IDL$ as unusable (R); results $< IDL$ are acceptable.
 - If the ICV/CCV %R is significantly lower than the lower control limit (LCL), qualify all associated data as unusable (R)
 - If the ICV/CCV %R are outside the acceptable limits, yet relatively close to the limits, qualify associated results as estimated (J if $> IDL$; UJ if $< IDL$ and the %R is below the LCL).

F. BLANKS

Evaluation

1. Review the raw data for all blanks and verify the results were accurately reported.
2. No contaminants should be found in the blanks (i.e. all blank results should be $< IDL$).
3. Positive blank results must not be corrected by subtracting off the blank value from the sample results.

Action

1. Sample results $\geq IDL$ but less than 5 times the maximum concentration found in any blank should be qualified as not detected (U).
2. Any blank with a negative result whose absolute value is $> IDL$ must be carefully evaluated to determine if the sample data is biased in any way.

G. LABORATORY CONTROL SAMPLES (EPA REFERENCE SAMPLES)

Evaluation

1. The laboratory control sample (LCS) serves as a monitor of the overall performance of the analysis including all preparation steps. Review the LCS and verify that the results fall within the control limits required. (If no limits noted, use 80-120% for the control limits.)
2. Check the raw data to verify the reported recoveries. Recalculate one or more recoveries (%R) using the following equation:

$$\text{LCS \%R} = \frac{\text{LCS Found}}{\text{LCS True}} \times 100$$

Action

1. If results are < IDL and the LCS recovery is above the UCL, the data are acceptable.
2. If the LCS recovery for any analyte falls within the range of 50-LCL, or > UCL, qualify results > IDL as estimated (J).
3. If results are < IDL and the LCS recovery falls within the range of 50-LCL, qualify the affected results as estimated (UJ).
4. If the LCS recovery results are < 50%, qualify the data for the affected analytes as unusable (R).
5. If a LCS was required and not run, use professional judgement to determine whether the data is estimated (J) or unusable (R).

H. LABORATORY DUPLICATES

Evaluation

1. Review the duplicate data and verify that the results fall within the criteria required. If no criteria exist, use the limits of 20% for the Relative Percent Difference (RPD) or \pm IDL if results are < 5x IDL (35 RPD or \pm 2x IDL if > 5x IDL for soils).
2. Review the data and verify that the duplicate analysis was not performed on a field blank.
3. Check the raw data and recalculate one or more RPD using the following:

$$RPD = \frac{|S-D|}{(S+D)/2} \times 100$$

where, S = Original sample value
D = Duplicate sample value.

Action

1. If duplicate analysis results for a particular analyte fall outside the appropriate control windows, qualify the results for that analyte in all associated samples of the same sample matrix as estimated (J).
2. If the field blank was used for duplicate analysis, carefully evaluate all other quality control data and use professional judgement in evaluating the data.

I. MATRIX SPIKES

Evaluation

1. Review the matrix spike data and verify that the results fall within the limits specified. If no criteria exists, use the limits of 75-125% for both water and soil matrices.
2. Check the raw data and recalculate the % Recovery of at least 10% of the data. Use the following equation to verify that the results were calculated correctly:

$$\%R = \frac{(SSR - SR)}{SA} \times 100$$

Where, SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added

3. Review the data and verify that the field blank was not used for the spike analysis.

Action

1. If the spike recovery is > 125% (or > UCL stated in the procedure) and the sample results are < IDL, the data is acceptable for use.
2. If the spike recovery is > 125% or < 75% (> UCL or < LCL stated in the procedure) and the sample results are > IDL, qualify the data for these samples as estimated (J).
3. If the spike recovery falls within the range of 30-74% (30-LCL) and the sample results are < IDL, qualify the data for these samples as estimated (UJ).
4. If any spike recovery results are < 30% and the sample results are < IDL, qualify the data for these samples as unusable (R).

5. In cases where more than one spike analysis was performed and one or more %R results were outside acceptable ranges, use professional judgement when qualifying the data. Matrix spikes should be performed on each sample matrix. Qualify data of similar matrix as the spiked sample if different matrices in the data package.
6. If the field blank was used for the spike analysis, carefully evaluate all other quality control criteria and use professional judgement in evaluating the data.

J. SAMPLE RESULT VERIFICATION

Evaluation

1. Examine the raw data to verify the analyte quantitation was calculated as stated in the procedure. Re-calculate a minimum of 10% of the sample results to verify results were calculated correctly.
2. Examine the raw data for anomalies such as baseline drift, negative absorbances (indicative of interferences), omissions, etc.
3. Verify there are no transcription or reduction errors (dilutions, percent solids, sample weights) on at least 10% of the samples.
4. Verify that all results fall within the calibrated range.

Action

1. If any discrepancies are found and can be corrected, make the corrections and note. Increase the level of quality control if necessary. If the discrepancy cannot be resolved by the reviewer, contact the data management coordinator. The laboratory or appropriate agency will be contacted and the information necessary to complete the evaluation will be requested. The project manager should be informed of the delay in the validation process. If any discrepancy remains unresolved, use professional judgement in qualifying the data.

K. FIELD DUPLICATES

Evaluation

1. Field duplicate samples measure field and lab precision and therefore the results may have more variability than lab duplicates. Review the field duplicate data and calculate the RPD. No criteria exists for evaluating the data.

Action

1. If the RPD is $> 50\%$, note in the narrative. Use professional judgement in qualifying any data. Review the raw data to verify that no reduction errors exist. This is to verify that the discrepancy is due to sampling techniques (sampling, preservation,

filtering, etc.) rather than laboratory error. Make notation of the discrepancy in the narrative.

L. OTHER (as specified in the procedure)

Evaluation

1. Review the procedure for any other quality control criteria not covered under the above sections. Examples of additional information required may be: ICP interference check samples, special AA quality control checks, distilled/digested blanks and standards, standardization, specific methodology requests for different concentration levels, etc..

Action

1. If specific quality control criteria were required and not followed, the project manager should be informed.
2. If the data was outside the acceptable ranges, use professional judgement in qualifying the data. Clearly identify your action and justification of the action in the narrative.

M. OVERALL ASSESSMENT FOR A CASE

1. It is appropriate for the reviewer to make professional judgements and express concerns and comments on the validity of the overall data package. This is particularly appropriate when several QC criteria are outside specifications. It is the reviewers responsibility to thoroughly document and explain all data validation qualifiers added to the data.
2. The following is a summary of data validation qualifier definitions which can be used in evaluating the data:
 - U The material was analyzed for, but was not detected above the level of the associated value. The associated value is either the sample quantitation limit or the sample detection limit.
 - J The associated value is an estimated quantity due to quality control criteria not being met.
 - R The data are unusable. The analyte may or may not be present.
 - UJ The material was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.
5. Once the data has been evaluated against all of the points described in part 4, the evaluator can prepare the data validation narrative.

The data validation narrative should be a summary of the data quality indicating any problems or deficiencies found with the data set. If

possible, on data qualified as estimated, note if the data is biased high or low. It is easiest to discuss each analyte separately. Refer to Figure 2 for the form to be used when writing the data validation narrative.

6. Record any data validation qualifiers (DVQ) to the left of the results on the analytical report. The laboratory qualifiers (LQ) will usually be recorded to the right of the result by the laboratory who performed the analytical work. A copy of the lab qualifier definitions should be supplied with the data. Data entry people need to be able to distinguish between DVQ and LQ, so note location of qualifiers if located other than above.
7. Record the sample ID and date sampled as noted on the Chain of Custody on the analytical report.
8. Highlight POSITIVE-HIT data with a highlighter.
9. Stamp all of the analytical reports "VALIDATED". A "VALIDATED" stamp can be obtained from the data management coordinator.
10. Sign and date the Data Validation Request Form.
11. A quality control review needs to be performed on the validated data package. The entire validated data package (including raw data, evaluator's notes and narrative) should be submitted to the QC reviewer for a final review.

Quality Control Review:

1. The purpose of this quality control review is to verify that the data were validated according to the guidelines above, that all data validation qualifiers were added and also that the narrative is complete.
2. Review the validated data package for completeness. The data package should contain the following:
 - Data Validation Request Form
 - Inorganic Data Validation Narrative
 - Validated Analytical Results
3. Verify that results exist for all samples and all analytes requested in the data package.
4. Review the data validation narrative with the validated results for consistency, i.e., if the narrative stated that a particular analyte was qualified, verify that the qualifier has been recorded with the associated data. Check approximately 10% of the qualifiers. If discrepancies are found, increase the level of QC checks.

5. Review the narrative; check that the data has been evaluated according to the quality control criteria specified in the QAPP.
6. Verify that all positive-hit data have been highlighted and that the sample descriptions and sampling dates have been recorded on every result page.
7. Verify that the reviewer has signed and dated the validated data package.
8. If no discrepancies exist, sign and date the Data Validation Request Form. If the data package requires clarification, review the raw data and/or obtain clarification from the reviewer.
9. Deliver the final validated data package to the Data Management Coordinator when the quality control review is completed.
10. Recycle any raw data. Raw data is not submitted with the final validated data package.

INORGANICS
DATA VALIDATION NARRATIVE

Site Name _____ Project # _____

SMO Case # (SAS #) _____ #Samples/Matrix _____

Laboratory _____ Hours for Review _____

SOW # _____ EPA Validation Guidelines # _____

Sample Numbers _____

Validated By _____ Date _____

Reviewed By _____ Date _____

SUMMARY OF REVIEW:

CAW/dlk/KJD
[dlk-601-22]
60721-MD